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VOLUME XX AUGUST 31, 1931 NUMBER 17

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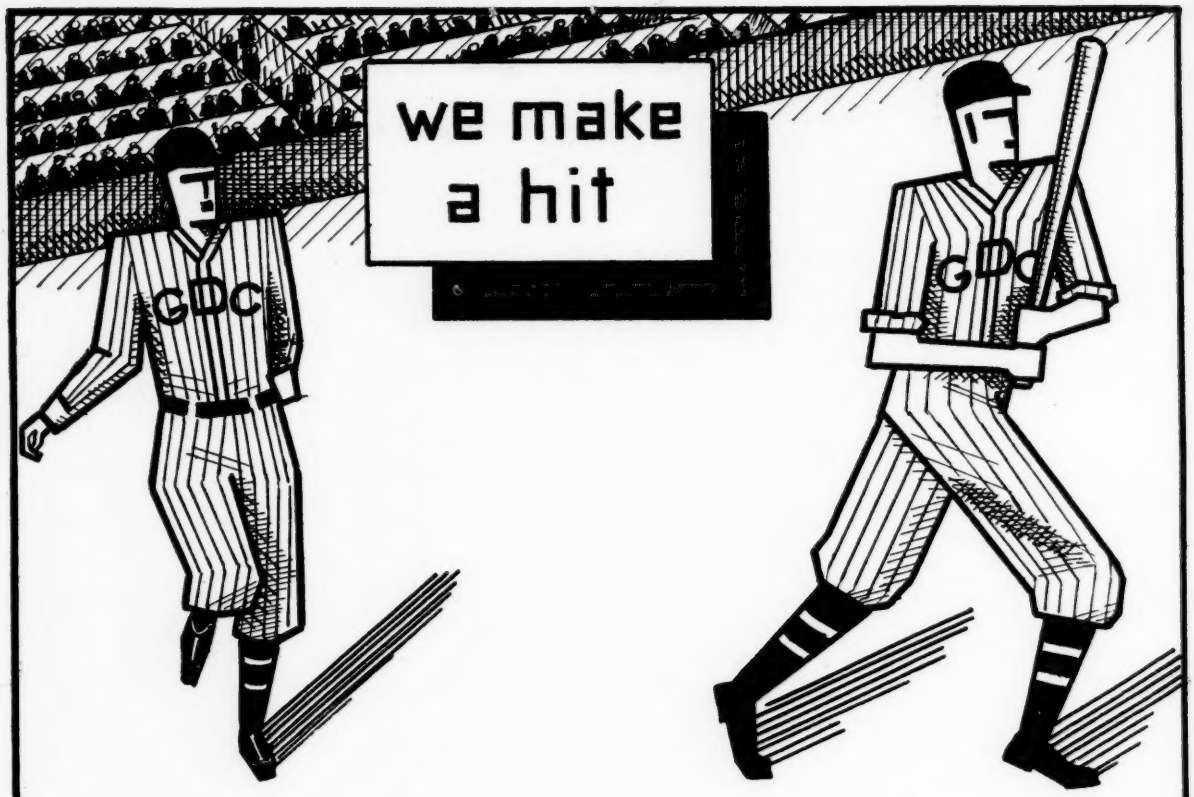

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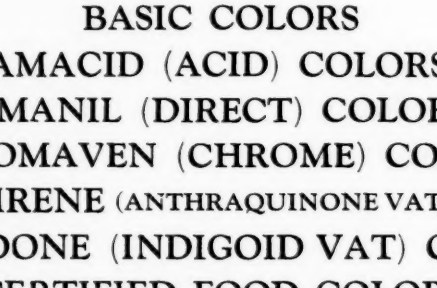

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Effects of Tension in Mercerization

By JOHN H. SKINKLE and RALPH E. HALE*

Cotton, which is the most used vegetable fiber that we have, has many valuable properties, one of which is the production of stronger and more lustrous yarn by the action of concentrated sodium hydroxide upon it. It is this action which is known as mercerization, that is to be studied quantitatively in this thesis, especially the effect of time and tension.

Quite a lot of work has been done upon the subject of mercerization, but most of the work has been done only qualitatively and what quantitative work has been done has been upon the effect of the strength of the sodium hydroxide. Most of the recent improvements of mercerizing are mechanical.

History

In 1844, Mercer was the first to recognize the fact that a strong solution of sodium hydroxide had an action on the cotton fiber. But it was not until 1889 when Lowe observed that by the application of tension an increase in luster was obtained, that the process became of great practical importance. The first demand for luster in cotton yarns came from the silk industry for use as warps in weaving.[1]

Industrial Processes

The technical process of yarn mercerization is as follows:

1. Preparation of the yarn.
2. Making up the set (in warp mercerization).
3. Preliminary boil out.
4. Impregnation or primary mercerization.
5. Washing or second mercerization.
6. Souring or neutralization.
7. Aftertreatment of the yarn.
8. Drying.

Before this treatment the yarns are very often gassed or singed due to the fact that if the fibers were left they would pass through without any tension, also they would cause the yarn to have a hazy outline instead of a clear cut one. When the yarn enters the boiling boxes it is better to use sulphonated oil than sodium carbonate, because of the fact that carbonate which may be carried over into the caustic has a tendency to cut down the mercerizing action. The sodium hydroxide boxes are usually three in number, sodium hydroxide enters the third, then flows over into the second, then into the first and from there it goes to the make up tank. The tension is applied before entering the sodium hydroxide boxes by differences in speed of the squeeze rolls. If there is too much weight on the rolls there is danger of chaffing. This tension is never removed until after the yarn has passed into the acid bath. The time of immersion in the commercial process is from one to five minutes.

The acid for the sour should not be added directly in this process due to the fact that there would be quite an evolution of heat and the mixing might not be thorough.[10] After the sour the yarn is washed first in one box of cold water, then in a second wash of from 90°-100° F., then a third of 120° F. A weak solution of ammonia at 120° F. is often used to neutralize any excess acid. Following this are two more hot washes, then a bath of a softener. After this a double squeezing is given to help make the drying easier. The drying may be accomplished with air drying or dry cans. The air driers produce the best results.[4]

Chemical Theory

When cold concentrated sodium hydroxide acts upon cellulose there is a marked evolution of heat indicating that a reaction has occurred chemically; if the alkali cellulose is then washed, the sodium hydroxide may be quantitatively removed. After mercerization without washing, the product is soluble in carbon bisulphide and a product is obtained that is soluble in sodium hydroxide solution, this is called viscose. A fundamental change has occurred in the cellulose molecule during the process. Since carbon bisulphide has no action on the pure cellulose there must have been an intermediate product formed. This argument is one of the strongest points of the chemical theory of mercerization.

*Presented by the junior author as a thesis for the degree of Bachelor of Textile Chemistry at the Lowell Textile Institute.

Colloidal Theory

Colloidal substances are composed of particles invisible under the microscope, but made up of many molecules. The larger the number of molecules to a particle the less is its chemical activity. According to this theory on mercerizing with sodium hydroxide the large particles of cotton are broken up into small particles and then left in that form when the sodium hydroxide is washed out. Thus with the spaces between them they occupy more space than they did before, and with the greater surface area they are able to have greater absorption of dyes and chemicals.[11]

Effect of Concentration of Sodium Hydroxide

The consensus of opinion in the literature is that mercerization begins at a concentration of 20° Tw. and reaches a maximum at from 40-50° Tw. Decrease in the mercerizing effect is much less above a concentration of 50° Tw. than it is below 40°.[11]

Hubner and Pope found by watching the progress of mercerization under a microscope that there was no effect up to 20° Tw. At 20° Tw. the fiber began to untwist, but no swelling of the fiber occurred until after the concentration had reached 35° Tw. After 40° Tw. had been reached untwisting and swelling occurred simultaneously. To get the maximum effect it must both swell and untwist.[11] It has been found from mercerization of a single hair that contraction begins with a sodium hydroxide strength of around 22° Tw. and when it reaches 60° Tw. the contraction has become very slow. The reason advanced why mercerization takes place during washing is that the sodium hydroxide becomes diluted to the optimum concentration during the washing process. It was observed that during washing the hairs had an additional loss in length and that at the same time they increased in diameter.[6]

Vieweg found that absorption of sodium hydroxide increased continuously with the concentration of the alkali. His data is insufficient, but shows two levels where absorption of sodium hydroxide remains constant for considerable changes of sodium hydroxide solution strength. (Fig. III.) These levels correspond to definite compounds and Vieweg's data is considered strong proof of the chemical theory of mercerization.[3]

Effect of Time

Gardner found that the action of sodium hydroxide was complete in one minute and that after five minutes, if the cotton was exposed to the air, it becomes tendered. In general practices two minutes is accepted as sufficient time.[4]

Effect of Temperature

Temperature plays an important part in obtaining good luster. Temperature necessary to obtain a satisfactory luster varies with the concentration of the sodium hydroxide bath. At low temperatures it is claimed that fine yarns will become transparent. At high temperatures better penetration is obtained, but the luster obtained is much inferior, it is also claimed that mercerization may be carried out at high temperature provided that the sodium hydroxide is sufficiently strong.[3]

It has been found that heat is developed by the action of sodium hydroxide on cotton and it has been measured and found to increase as the strength of the sodium hydroxide increases. It was also shown that the heat produced aided in the penetration of the sodium hydroxide.

The cause of luster is not the mercerization itself but physical effects resulting from mercerization. Luster depends upon the reflection of light and therefore is due to the surface characteristics of mercerized cotton yarn made up of a large number of fibers with round smooth surfaces which cause the reflection of light in parallel rays. The degree of twist is also a very important factor.[2]

In the application of tension to obtain luster the tension may be applied before immersion in the sodium hydroxide or after the maximum shrinkage occurred and before washing, or after impregnation and washing loose, to stretch to its original length.[3] To obtain maximum luster the tension must be sufficient to bring the yarn back to its original length. Hard twisted yarns require more tension in order to obtain the same luster as the loosely twisted yarn. Fibers projecting from the surface will not obtain luster, that is why many yarns are gassed or singed before mercerizing.[4]

It must be kept in mind that all yarns and fabrics will not produce the same luster. Luster depends on the staple length of the cotton, shorter fibers need more, and more twist will mean that impregnation will not be as fast nor will the fibers be parallel to the length of the yarn.[3]

The results of Herbig on the effect of tension may be summarized as follows:

1. Mercerization without tension gives no luster while even a small amount of tension gives some increase in luster.

2. Maximum luster is obtained when the tension is sufficient to bring the material back to its original length and any increase in tension above this does not increase the luster.

3. The luster obtained by impregnating and washing under tension is the same as impregnating loose and washing under tension, but more tension is required in the second case.

4. By impregnating and washing loose then stretching the luster is much inferior, and the tension required is very great.

5. Cotton yarn made of long staple cotton receives the highest luster.

Previous work by Lindsly[9] has shown:

1. Shrinkage decreases regularly but not proportionally with increase of tension.

2. All shrinkages approach separate points.

3. Final wet shrinkage becomes zero at a tension of 1800 grams under the condition employed.

4. Final dry shrinkage becomes equal to maximum shrinkage when mercerizing at a tension of 800 grams.[9]

Experiments on yarn mercerization are complicated

by a large number of variables and while it is possible to see the one yarn has come out better than the other, it is very hard to trace the cause. It has been found that control of twist, tension and uniformity of yarn must be exercised. The latest developments in mercerization have nearly all been improvements in machinery rather than scientific discoveries.[8]

PHOTOGRAPHIC METHOD OF MEASURING LUSTER[13]

The luster of a doubled yarn depends upon its doubling twist. The yarn which had had its twist altered by the addition and removal of doubling twist, were arranged in ascending order of twist on a black background and a beam of a light from a projection camera was focused upon it. Illumination was secured by correctly centering the source of light from a 500 candle power lamp with reference to the lantern condenser. Results showed they had either an extremely high or an extremely low doubling twist. The method was greatly improved by introducing into it a neutral wedge in the position usually occupied by the lantern slide in such a manner that the wedge causes the light intensity to be varied laterally. The specimens are illuminated by a beam of light that varies laterally in intensity. The distance in which it is invisible in the photograph is a measurement of its luster. The distance to which it will extend into the shadowed portion will depend somewhat on the regularity of the yarn that is photographed.[13]

EFFECT OF HEAT ON COTTON[14]

Forman and Meale have shown that drying a specimen of cotton at 110° C. for twenty-four hours reduced its viscosity in cuprammonia solution from 71 to 45[14]. This seems to indicate degradation of cotton by heat and may explain anomalous results of mercerization on bone dried and air dried cotton which the authors detected in the course of this experimental work.

APPARATUS USED

The instrument (Fig. I) that was used to obtain the results was developed by Skinkle and Lindsly when the latter was working on his thesis several years ago.

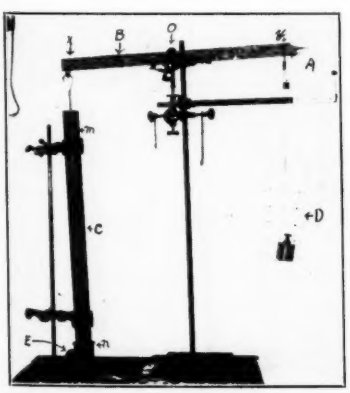


Fig. I

It consists of the pipe C having a plug E at the bottom which can be opened quickly and get the solution out rapidly. The arm B is the same distance from X to O as it is from Y to O, and the arm balance with the basket D suspended at one end and the hook at X on the other. The basket D is used to hold the weights that are employed to obtain the desired tension. After the yarn has been suspended between the hooks at M and N and the desired tension has been applied the table A is set at "O". The sodium hydroxide can then be poured in and the shrinkage registered on the scale.

Due to the fact that the pointer on one balance arm made that arm longer than the other, readings of shrinkage had to be multiplied by a factor of 0.78 to give the true shrinkage of the skein. The shrinkage in percentage was obtained by taking the factor 0.78, multiplying by 100 and dividing by 18 which was the original length of the skein. The result of this gave the constant factor 4.33, by which all shrinkages were multiplied to get percentage.

PREPARATION OF CARBONATE FREE SODIUM HYDROXIDE

To prepare the mercerizing solution of sodium hydroxide, C. P. sodium hydroxide was obtained and dissolved in distilled water to the amount of 265 grams of sodium hydroxide to one liter of water. This is allowed to stand at least twenty-four hours and after that time it is titrated to find the amount of sodium hydroxide and also the sodium carbonate. The titration was first carried out with phenolphthalein to the end point, then continued with Methyl Orange.

The amounts of sodium hydroxide and sodium carbonate are calculated and then enough of a saturated solution of Barium Hydroxide is added to precipitate the Carbonate out of the solution. Thus the Barium Carbonate will precipitate and settle out. After standing over night the clean solution is decanted off and diluted until a concentration of 250 grams per liter is reached.

SAMPLE CALCULATION FOR THE CARBONATE FREE SODIUM HYDROXIDE

Phenolphthalein Indicator

5 cc. sample = 32.05 cc. of 1.316 N acid

With Methyl Orange in same bath

0.23 cc. of 1.316 N acid

0.23 x 2 = 0.46 cc. of 1.316 N acid required to neutralize the carbonate

32.05 — 0.23 = 31.82 cc. 1.316 N acid to neutralize the sodium hydroxide

$$31.82 \times 1.316 \times .040 \times \frac{1000}{S} = 335.0 \text{ grams per liter of sodium hydroxide}$$

$$0.46 \times 1.316 \times .053 \times \frac{1000}{S} = 3.21 \text{ grams of sodium carbonate per liter}$$

4 liters of solution

4 x 3.21 = 12.84 g. total amount of sodium carbonate

$$Na_2CO_3 + Ba(OH)_2 = BaCO_3 + 2\ NaOH$$
$$106 \qquad 171 \qquad\qquad 80$$

$$12.84 \times \frac{171}{106} = 20.75 \text{ grams of } Ba(OH)_2 \text{ required to precipitate the sodium carbonate}$$

10 cc. sample of the Barium Hydroxide solution was titrated with .1316 N acid.

40.05 cc. .1316 N acid Methyl Orange Indicator 40.05 x

$$.1316 \times .0855 \times \frac{1000}{10} = 45.10 \text{ g. of } Ba(OH)_2 \text{ per liter.}$$

$$\frac{20.75}{45.10} \times 1000 = 461 \text{ cc. of the solution required to precipitate the sodium carbonate.}$$

During this reaction sodium hydroxide is formed thus strengthening the solution more.

$$12.84 \times \frac{80}{106} = 9.70 \text{ grams of sodium hydroxide formed.}$$

Volume after addition of $Ba(OH)_2$ solution—4,461 cc.

335.0 x 4 = 1340 grams of sodium hydroxide

1340 grams + 9.70 grams = 1349.70 grams of sodium hydroxide. Total amount in solution.

$$\frac{1349.70}{4461} = 0.303 \text{ g. of sodium hydroxide per cc.}$$

Desired—0.25 g. of sodium hydroxide per cc.

$$\frac{1349.7}{.25} = 5{,}378.8 \text{ cc. the volume should be to have 0.25 g. sodium hydroxide per cc.}$$

5,378.8 — 4461 = 917.8 cc. of water to be added to make the concentration approximately 250 grams per liter.

24.10 cc. of 1.316 N acid with phenolphthalein Indicator

At neutral point when Methyl Orange was added. Thus it showed there is no carbonate present.

$$24.10 \times 1.316 \times 0.40 \times \frac{1000}{5} = 254.0 \text{ g. sodium hydroxide per liter.}$$

Detailed Description of a Complete Mercerizing Run

The 2/20's cotton was boiled out with 2% sodium carbonate for one hour and then given a good rinse and dried. It was wound off on a 36 inch reel into skeins of 80 yards in length and then placed in a conditioning oven at a temperature of 105° C for about one and a half hours. This was done at first in order to weigh the skeins before and after mercerizing but later the weighing was discontinued but the bone drying was still carried out so that the conditions would always be the same. The skein was then suspended between the hooks m and n (Fig. I) and then the weight necessary to give the proper tension (in this case 1000 grams) was placed in the basket O. Two hundred and fifty cc. of sodium hydroxide solution was taken and its temperature is adjusted at 25° C before starting. The scale must be adjusted so that the pointer is at the zero point before the solution is poured in.

Readings were taken every thirty seconds until the maximum shrinkage had been reached and maintained until constant readings were obtained. During the first few runs a 5 cc. sample of solution was taken at every reading but this was discontinued due to the fact that the apparatus was not adapted to the taking of representative samples.

When the maximum shrinkage had been reached the solution was rapidly drawn off and water at 25° C was poured in. Stretching took place and continued for some time, finally becoming constant. The first wash water was rapidly removed and 250 cc. more of water at 25° C was added and readings were taken every minute until constant readings resulted. This washing was continued until a fresh lot of water did not give a variation on the scale. The skein was then removed, given a wash, and then placed in an acid bath made up of 10 cc. of 6 normal sulfuric acid to 500 cc. of water. It remained in this bath for 10 minutes, was then removed and given a thorough wash, then placed in an ammonium hydroxide solution of 10 cc. of 6 normal ammonium hydroxide in 400 cc. of water for 10 minutes, and was finally given a thorough wash and bone dried.

Measurement of the Breaking Strength

The breaking strength of the cotton yarn was measured on a machine made by Louis Schopper of Leipzig. The skeins were placed in the testing laboratory the day before the runs were carried out so that they would be at the proper condition when they were tested. The conditions were a temperature of 70° F. and 65% relative humidity. The speed of the moving jaw was fixed at 12 inches per minute and the length between the jaws was 10 inches. On each skein the individual yarns were picked out and twenty breaks were made on each skein. Those which broke at the jaws were discarded.

Measurement of the Count

The count was measured by taking one half of the skein, which measured over 14 inches, and cutting off exactly ten inches using the same tension each time. Then from these there was counted out enough strands to make the weight as near as possible to one gram (it was desired to use the same sample for dyeing tests). The number of strands taken was recorded and then the skein was placed in a conditioning oven to obtain a bone dry weight. The weight was recorded and then the count was calculated as follows:

Sample Calculation

Weight 0.9826 grams

Strands 65 (10 inches long)

$$\frac{65 \times 10}{12 \times 3} = 18.05 \text{ yards}$$

$$0.9826 \text{ x } 1.065 = 1.048 \text{ grams of cotton with 6.5\% regain}$$

$$\frac{18.05}{1.048} = \frac{X}{454} \qquad X = 7820 \text{ yards} \qquad \frac{7820}{840} = 9.3 \text{ Hanks}$$

or since the yarn is 2 ply $\frac{18.6}{2}$

Measurement of the Dye Absorbed

The sample from the determination of the count were all dyed with 1% Erie Fast Scarlet 4 BA and 20% Sodium Chloride for exactly one hour. The volume of the bath was kept constant all the time at 75 times the weight of the samples.

The material was dyed in 250 cc. Erlenmeyer flask with a five foot air condenser of one-half inch glass tubing. The volume of the dyebath was around 75 cc. but varied each side of it according to the amount that the sample varied from one gram.

The samples were removed by means of a hook as quickly as possible when the time limit was reached and worked a few times in four baths of water. This was done identically with each sample so that the washing

would be the same in all cases. After the solution had cooled down to room temperature it was compared in a Dubosco colorimeter with a sample of the dye solution as made up before dyeing and six readings were taken and averaged to find the ratio between them.

In making up the dye solution it was desired to make up the solution so that for exactly 1 gram samples 75 cc. could be used.

SAMPLE CALCULATION

1% dye 1 gram sample required 0.01 g dye
20% salt 0.20 g salt

$$\frac{1000 \times .01}{75} = 0.133 \text{ g of dye per liter}$$

$$\frac{1000 \times .20}{75} = 2.666 \text{ g salt per liter}$$

For example if the sample weighed 1.035 grams, then $1.035 \times 75 = 77.6$ cc. of dye solution would be required for that sample.

The ratio between the standard and the sample was obtained by dividing the depth of the standard by the depth of the sample. From the ratio the milligrams of dye absorbed per gram of bone dry cotton can be calculated.

Dye bath = 0.133 grams per liter
Weight (W) = 0.9881 grams
Cubic centimeters of dye taken (D) = 73.1 cc.
Ratio of dyebath to initial (R) = .209
The following formula was worked out

$$\frac{.133 \text{ D } (1 - \text{R})}{\text{W}} = \text{Mg of dye per gram of bone dried cotton.}$$

$$\frac{.133 \times 73.1 \times (1 - .209)}{.9881} = 7.78 \text{ Mg of dye absorbed per gram of bone dried cotton.}$$

MEASUREMENT OF LUSTER BY PHOTOGRAPHIC MEANS

This method was originally developed by Adderly as already described. He used a neutral wedge to obtain his laterally reflected light.

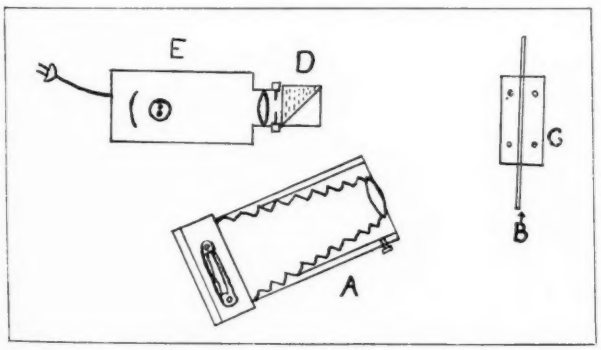


Fig. II
A—camera. B—card with sample, C—stand, D—hollow glass prism, E—ribbon filament tungsten lamp.

It was impossible for the author to obtain a neutral wedge but instead an attempt was made to utilize a hollow glass prism containing a red dyestuff so that the light passing through it would vary greatly in intensity. Arrangement of the apparatus used is shown in Fig. II.

The light B is so constructed that it will give a strong source of light, well centered. The light passes through C which is a hollow glass prism containing the red dyestuff solution.

On card D, which was a flat black card, there was placed (about one-eighth of an inch apart) strands of cotton which had been mercerized with varying tensions, also a sample of rayon.

The camera was focused and exposed on the card. As the strength of the dyestuff solution had to be quite strong the length of exposure required was around 15 minutes using panchromatic plates.

No satisfactory results were obtained, and as time was lacking this part of the work was discontinued.

DISCUSSION OF EXPERIMENTAL PROCEDURES

It is very essential that carbonate free sodium hydroxide be obtained, since it is known that carbonate in the bath slows down the mercerization effect. Also great care must be taken that it is not left exposed to the air because it is very likely to pick up carbon dioxide very rapidly, therefore from time to time it is well to check up on the carbonate content, and to seal over the stopper of the bottle containing the sodium hydroxide with melted paraffin.

It is essential that enough yarn be boiled out at once for a complete set of runs and also for a blank, so as to eliminate as much as possible the variation of the yarn entering the mercerizer.

On the first few runs samples of solution were taken along with every reading and titrated. It was hoped that there could be found something in regard to the amount of sodium hydroxide that is absorbed by the cotton, but it was found that representative samples could not be obtained and the procedure was abandoned.

In the first few mercerizing runs that were made a thermometer was placed in the bath to try and record the rise in temperature as the action proceeded. It was found that temperature changes were too small to be recorded on an ordinary thermometer.

After mercerizing and in neutralizing it is very essential that the time and treatment be the same due to the fact that variation here will cause variation in strength and dyeing properties. The ends of the skein where they have been drawn against the hook will not be mercerized. This effect increases as the tension goes up. It is advisable after neutralizing to cut these ends off to avoid the chance of getting them included in a test sample later on.

On the first few runs, weighings of the skein before and after mercerizing were made with the idea of seeing whether there was any appreciable gain in weight, as a matter of fact, all of the skeins showed a small loss in weight, but this loss showed no regularity and the measurement of it was discontinued.

One set of runs was made, using air dried instead of bone dried yarn. The results obtained showed that this

Sample Run and Calculation

Tension 500 grams. Temperature at start 25° C.
5 cc. sample placed in 25 cc. standard acid

Time	Shrinkage inches		Factor		Percent	Base cc.	Acid cc.		Factor		Grams per liter
½	0.0	x	4.33	=	0.0%						
1	0.20	x	4.33	=	0.87%	1.20	24.31	x	102.7	=	250
1½	0.45	x	4.33	=	1.94%						
2	0.75	x	4.33	=	3.28%	0.55	24.68	x	102.7	=	254
2½	0.85	x	4.33	=	3.66%						
3	1.0	x	4.33	=	4.33%	1.10	24.37	x	102.7	=	251
3½	1.15	x	4.33	=	5.00%						
4	1.30	x	4.33	=	5.61%	0.40	24.77	x	102.7	=	255
4½	1.40	x	4.33	=	6.06%						
5	1.50	x	4.33	=	6.50%	0.50	24.71	x	102.7	=	254
5½	1.55	x	4.33	=	6.72%						
6	1.60	x	4.33	=	6.95%	0.40	24.77	x	102.7	=	255
6½	1.70	x	4.33	=	7.34%						
7	1.80	x	4.33	=	7.78%	0.80	24.55	x	102.7	=	252
7½	1.80	x	4.33	=	7.78%	0.85	24.52	x	102.7	=	252
					Wash I 10 cc. of standard acid used						
8	1.60	x	4.33	=	6.95%	15.55	1.04	x	10.27	=	10.7
9	1.55	x	4.33	=	6.72%	16.75	0.58	x	10.27	=	5.9
10	1.50	x	4.33	=	6.50%	16.85	0.52	x	10.27	=	5.4
11	1.45	x	4.33	=	6.28%	16.80	0.55	x	10.27	=	5.7
12	1.40	x	4.33	=	6.06%	16.75	0.58	x	10.27	=	5.9
					WASH II						
13	1.25	x	4.33	=	5.44%	17.15	0.35	x	10.27	=	3.6
14	1.15	x	4.33	=	5.0 %	17.15	0.35	x	10.27	=	3.6
15	1.15	x	4.33	=	5.0 %	17.05	0.40	x	10.27	=	4.1
					WASH III						
16	1.10	x	4.33	=	4.75%	17.25	0.29	x	10.27	=	3.0
17	1.05	x	4.33	=	4.55%	17.20	4.32	x	10.27	=	3.3
18	1.05	x	4.33	=	4.55%	17.20	0.32	x	10.27	=	3.3
					WASH IV						
19	1.00	x	4.33	=	4.33%	17.20	0.32	x	10.27	=	3.3
20	1.00	x	4.33	=	4.33%	17.20	0.32	x	10.27	=	3.3
					WASH V						
21	0.95	x	4.33	=	4.11%	17.25	0.29	x	10.27	=	3.3
22	0.95	x	4.33	=	4.11%	17.25	0.29	x	10.27	=	3.0

Summary of Data

Tension Grams	Percent Max. Shrinkage	Percent Final Shrinkage	Percent Max. Stretch	Time (Min.) to Max. Shrinkage	Time (Min.) to final Shrinkage	Time (Min.) to Max. Stretch	Time (Min.) to cross O axis	Length of time of first wash	Time to Max. shrinkage—Time to Max. stretch	Time to final shrinkage—Time to max. stretch
20	24.90	18.60	0	11	15	0	0	6	11.0	15.0
500	7.78	4.11	0	7½	15	0	0	5	7.5	15.0
1000	5.52	1.55	–0.87	12½	17	½	1–1½	6	12	16.5
2000	2.17	–1.22	–91	15½	25	1	3–3½	9	14.5	24.0
					SET II					
100	17.6	12.1	0	9	13	0	0	4	9.0	13.0
500	8.23	3.90	–0.87	9	26	½	½–1	9	8.5	25.5
1000	6.07	2.25	0	10½	20	0	0	7	10.5	20.0
1250	4.98	1.30	–0.43	13	26	½	1	10	12.5	25.5
1500	4.72	0.74	–0.43	14	22	½	1–1½	9	13.5	21.5
1750	3.47	0.09	–0.43	16	26	½	1–1½	12	15.5	25.5
2000	2.08	–1.17	–1.08	16	30	1	4	15	15.0	29.0
					SET III					
2000	2.60	–0.91	–1.08	18	29	½	3½	17	17.5	28.5
2500	1.82	–0.87	–0.43	17	30	½	3	17	16.5	29.5
3000	1.73	–1.21	–0.35	19	32	½	2½	16	18.5	31.5
3500	1.26	–1.30	–0.43	21	24	1	4	14	20.0	23.0

Tension Grams	Percent Max. Shrinkage	Percent Final Shrinkage	Percent Max. Stretch	Time (Min.) to Max. Shrinkage	Time (Min.) to final Shrinkage	Time (Min.) to Max. Stretch	Time (Min.) to cross O axis	Length of times of first wash		
1750	3.30	–0.35	–0.87	16	27	½	2½	12	15.5	26.5
2000	3.90	0.43	–0.35	19	26	½	1½	13	18.5	25.5
2000	3.90	0.09	–0.43	24	30	½	2	15	23.5	29.5
2250	3.04	–0.34	–0.35	23	32	½	2	17	22.5	31.5
2750	1.30	–1.69	–1.08	17	24	½	5	10	16.5	23.5
3250	1.30	–1.43	–0.43	20	32	½	4	15	19.5	31.5
					SET III					
1750	2.60	–0.95	–1.08	13	31	½	2–2½	14		
2000	3.03	–0.43	–0.13	20	41	½	½–1	18		
2250	2.60	–0.43	–0.22	16	36	½	½–1	18		
2500	2.17	–0.87	–0.26	19	35	½	1–1½	18		
2750	2.17	–1.30	–0.17	18	32	½	1½	16		
3000	1.30	–1.35	–0.26	20	38	½	2½	20		
3500	1.21	–1.56	–0.22	22	35	½	2½	18		

Tension Grams	Shrinkage: Percent Max. Shrinkage	Percent Final Shrinkage	Percent Difference in Shrinkage	Percent Change in Shrinkage	Reading (Grams)	Strength: Blank	Ratio	Dyeing: Mg of dye adsorbed gram bone dry cotton	Blank	Ratio
20	34.90	18.60	6.30	25.	813	636	1.28	8.03	6.52	1.23
500	7.78	4.11	3.67	47.	599					
1000	6.39	2.42	3.97	62.	738		1.16	8.16		1.25
2000	3.08	–1.31	3.39	110.	698		1.10	8.05		1.24
					SET II					
100	17.60	12.10	5.50	31.	773	623	1.24	8.40	6.57	1.28
500	9.10	4.77	4.33		763		1.23	8.16		1.25
1000	6.07	2.25	3.82	63.	667		1.07	8.12		1.24
1250	5.41	1.73	3.68	68.	724		1.16	8.07		1.23
1500	5.15	1.17	3.98	77.	741		1.19	7.80		
1750	3.90	0.52	3.38		732		1.18	8.10		1.23
2000	3.16	–0.09	3.25	103.	701		1.13	8.05		1.22
					SET IV					
2000	3.68	0.17	3.51	95.	792	712	1.11	8.34	6.69	1.25
2500	2.25	–0.43	2.69	119.	771		1.08	8.04		1.20
3000	2.08	–0.86	2.94	148.	763		1.07	8.09		1.21
3500	1.69	–0.86	2.56	151	786		1.10	8.10		1.21
					SET V					
1750	4.17	0.52	3.65	88.	756	635	1.19	7.64	6.55	1.17
2000	4.25	0.78	3.47	82.	841		1.33	7.84		1.20
2000	4.33	0.52	3.81	88.0	717		1.13	7.79		1.19
2250	3.39	0.01	3.38	100.	719		1.13	7.91		1.21
2750	2.38	–0.61	2.99		690		1.09	7.78		1.19
3250	1.73	–1.00	2.73	158.0	702		1.11	7.40		1.13

Shrinkage—(Continued)

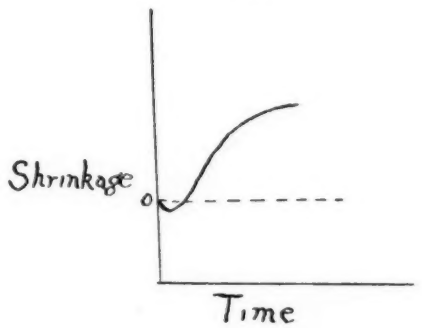
Tension Grams	Percent Max. Shrinkage	Percent Final Shrinkage	Percent Difference in Shrinkage	Percent Change in Shrinkage
1750	3.68	+0.13	3.55	97.
2000	3.16	–0.30	3.46	109.
2250	2.81	–0.21	3.03	108.
2500	2.43	–0.61	3.04	125.
2750	2.34	–1.13	3.47	148.
3000	1.56	–1.11	2.67	171.
3500	1.43	–1.34	2.77	193.

Tension Grams	Count: Reading	Blank	Ratio	Strength Constant: Reading	Blank	Ratio
20	13.84	18.20	0.76	11,270	11,600	0.97
500						
1000	16.94		0.93	12,500		1.08
2000	18.50		1.02	12,292		1.11
			SET II			
100	14.96	47.50	0.86	11,550	10,900	1.06
500	16.24		0.93	12,400		1.14
1000	17.10		0.98	11,410		1.05
1250	17.62		1.01	12,750		1.17
1500						
1750	18.14		1.04	13,280		1.22
2000	18.14		1.04	12,720		1.17
			SET IV			
2000	17.50	17.60	0.99	13,850	12,500	1.11
2500	17.48		0.99	13,480		1.08
3000	18.52		1.05	14,150		1.14
3500	18.82		1.07	14,800		1.19
			SET V			
1750	18.00	17.70	1.02	13.600	11,150	1.22
2000	16.04		0.90	13,520		1.21
2000	17.70		1.00	12,700		1.14
2250	18.60		1.05	13,380		1.20
2750	19.68		1.11	13,520		1.21
3250	18.98		1.07	13,310		1.19

treatment appreciably affected the results, but due to lack of time this field of investigation had to be left unfinished. In all other runs, therefore, bone dried yarn was used.

Deductions Drawn From the Curves

1. Examination of Figures IV-VII show that the variation of shrinkage with time in caustic is in general as follows:



This indicates that mercerization begins with simple wetting, and is then followed by absorption of the caustic up to a certain definite value.

2. The effect of water on the caustic-saturated yarn is at first rapid but eventually reaches a constant value.

3. The greater the tension on the yarn, the longer the time required to reach equilibrium following a minimum at 500 grams (Fig. VIII).

4. The greater the tension, the longer the time required for the wash water to come to equilibrium although there is apparently no difference in the number of washes required.

5. The total time for mercerization (including caustic treatment and washes) is practically constant above 2000 grams. (Fig. IX).

6. Both the maximum and final shrinkage drops rapidly at first with increasing tensions but finally becomes almost constant. (Fig. XI).

7. The final shrinkage becomes zero at about 2000 grams tension. This is the point above which, according to Herbig, there is no further advantage to be gained by tension. (Fig. XI).

8. The percentage of the total shrinkage recovered on washing is in straight line relationship to the tension and

is equal to 100% at 2000 grams. (Herbig's point). (Fig. XII).

9. In Fig. XII, it may be seen that the data obtained by a single run on air dried yarn, when plotted seems to intersect the other line at about 2000 grams. This however, should be checked further by runs before any conclusions may be drawn.

10. The ratio of dye absorbed by mercerized yarn to dye absorbed by unmercerized yarn is a maximum at low tensions and then decreases, becoming practically constant at about 2000 grams (Herbig's point). (Fig. XIV).

11. The ratio of the strength of mercerized to unmercerized cotton decreases with increasing tension to a minimum at about 1250 grams, rises to a maximum at 1600 grams and decreases to another minimum at 2750 grams. This is in agreement with the work of Skinkle and Lindsly, since their data did not continue to as high a tension as this work. It may be noted that above 2000

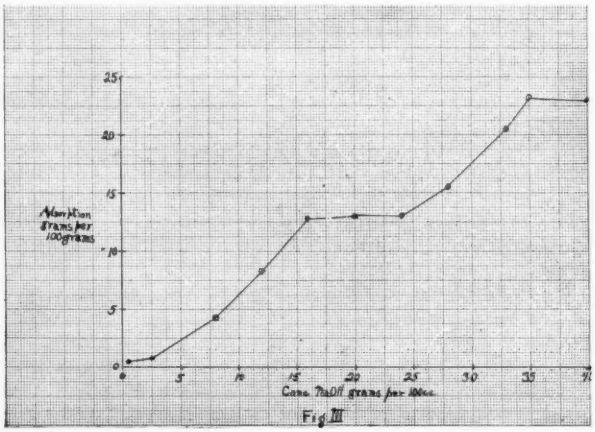


Fig III

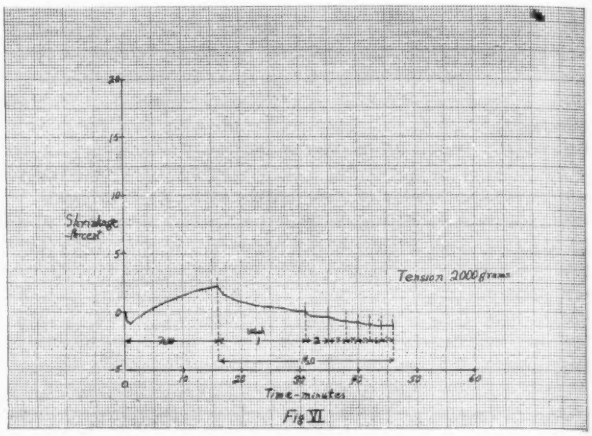


Fig VI

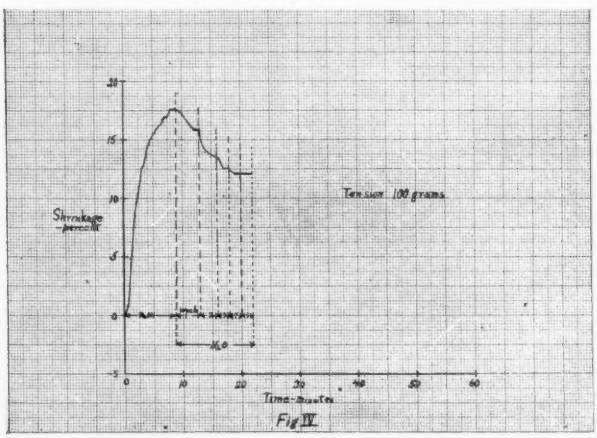


Fig IV

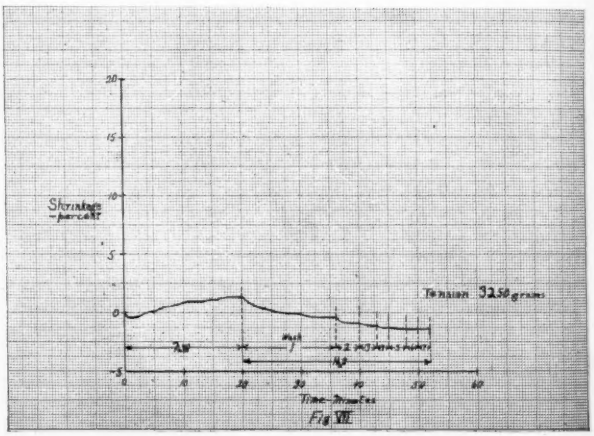


Fig VII

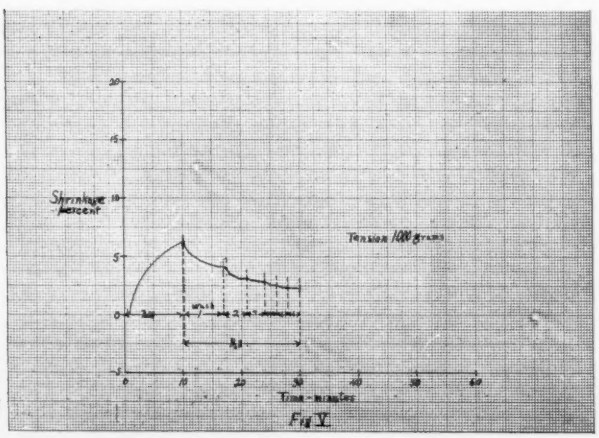


Fig V

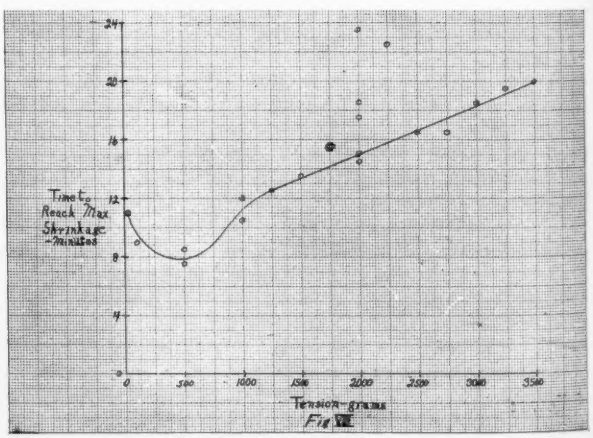


Fig VIII

grams the change in strength ratio while definite is not great. (Fig. XIII).

12. The count or size of yarn increases rapidly at first and then slowly with increasing tension. (Fig. XV).

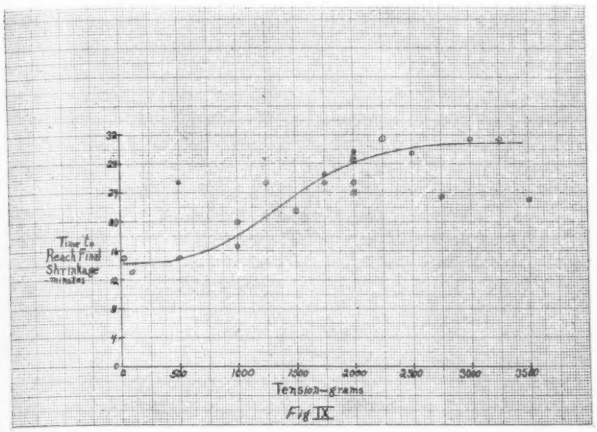


Fig IX

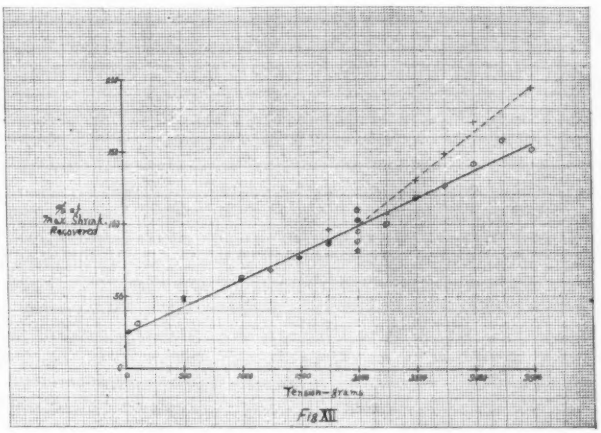


Fig XII

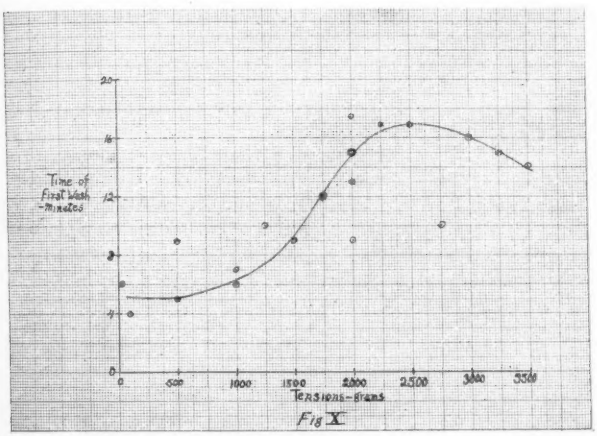


Fig X

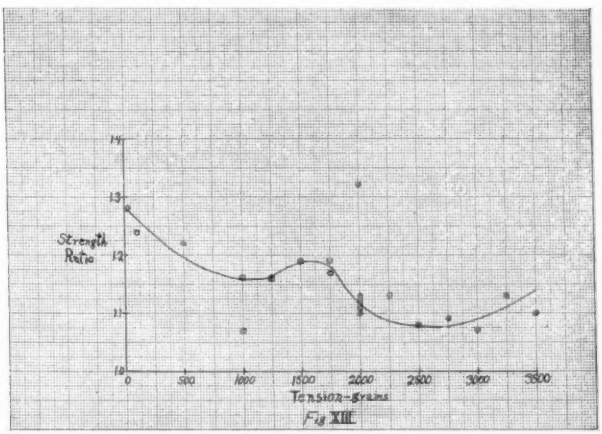


Fig XIII

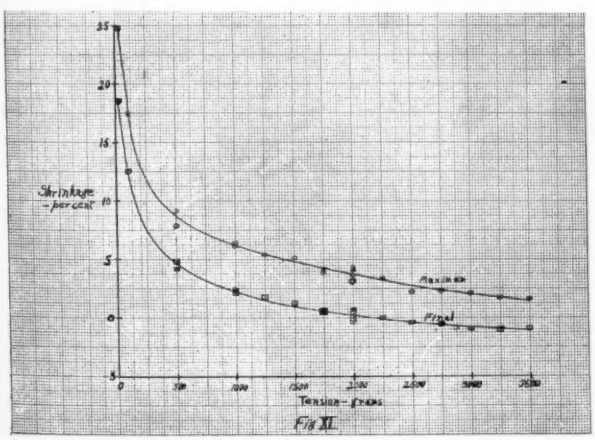


Fig XI

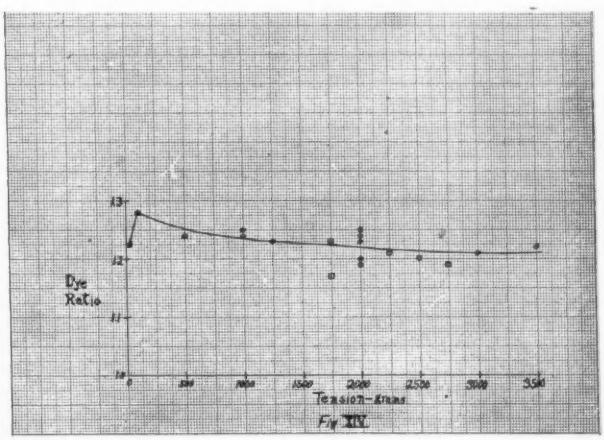


Fig XIV

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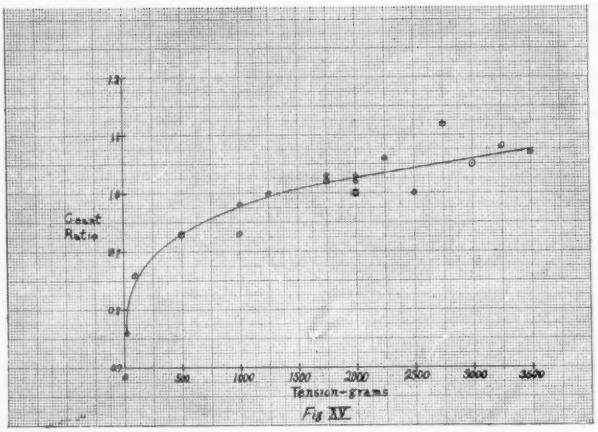

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CONCLUSION

1. The tension applied has considerable effect upon the rate of absorption of the caustic and the rate of washing of the yarn.

2. The amount of shrinkage recovered upon washing is directly proportional to the tension applied.

3. Any tension above that necessary to regain completely the shrinkage of the yarn in the caustic gives no appreciable advantage in the properties of the finished yarn. This is confirmation of the statement of Herbig above, except that Herbig speaks of luster which was not measured in this work.

Hercules Changes

A number of changes in the organization of the naval stores department of the Hercules Powder Company were announced recently by L. N. Bent, general manager of the department. J. E. Lockwood, formerly director of sales, is now assistant to the general manager. Arthur Langmeier, formerly assistant director of sales, becomes director of sales. George C. O'Brien, formerly manager of the Chicago office, is now assistant director of sales. Jesse Gibson, sales manager, assumes charge of Wilmington branch office sales. Arthur H. Sanford, formerly manager of Wilmington branch office, will do special sales work under the direction of Mr. Gibson.

New Patents

Method of Producing or Developing Azo Dyes. (By causing an alkali metal compound of a nitrosamine derived from p-nitranilin to react with a coupling component in the presence of sodium bicarbonate.) Hans Theodor Bucherer, of Charlottenburg, Ger., assignor to General Aniline Wks., Ins., of N. Y., N. Y., No. 1,815,965, July 28, 1931.

Indigoid Vat Dyestuffs. (Dye animal and vegetable fibers grey shades, also suitable for printing purposes, possessing good fastness properties to light and exposure; obtained by heating 5-chloro-isatine with phosphorus pentachloride and chloro-benzene.) Norbert Steiger, of Frankfort-on-the-Main, Ger., assignor to General Aniline Wks., Inc., of N. Y., N. Y., No. 1,816,660, July 28, 1931.

The Weighting of Silk

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CHAPTER II. THE DEVELOPMENT OF PRESENT DAY WEIGHTING PROCEDURE

(Photographs and sketches printed by the courtesy of the Van Vlaanderen Machine Company of Paterson, New Jersey.)

This is the second in a series of articles dealing with the weighting of silk. Chapter I appeared in the August 17th issue of the AMERICAN DYESTUFF REPORTER and was devoted to the historical consideration of the subject. The third chapter will deal with the chemical analysis and control as applied to the weighting baths and also with the analysis of weighted silks. The fourth chapter will take up the relative durability of weighted and unweighted silk and discuss the methods which have been proposed to increase the durability of weighted silks.

ESSENTIALS OF THE PROCESS

IN SPITE of the numerous patents for the weighting of silk which have been described in the previous chapter and the large variety of chemicals which have been suggested for this purpose, there has been surprisingly little change in the practical working formula which has been in use for a great many years. In fact, over 90% of the present day silk weighting is accomplished with the combination of tin, phosphate and silicate which was suggested by Bierman prior to the beginning of this century.

The mechanism of this process is very simple, although the chemical reactions which take place are so complex that they are not completely understood even now. Briefly, the silk is first immersed in a solution of stannic chloride which contains a small percentage of free hydrochloric acid. A certain amount of the stannic chloride is absorbed by the silk and the tin is then fixed on the fiber by the simple expedient of washing in clear water whereby the soluble stannic chloride is hydrolized to the insoluble stannic acid. After washing the silk is transferred to a slightly alkaline bath containing disodium phosphate whereby a complex tin-phosphate compound is formed. The alternate treatments with stannic chloride and sodium phosphate may be repeated a number of times and then the silk is finely immersed in a solution of sodium silicate which results in a further increase in weight by the formation of a still more complex tin silico-phosphate. A still higher weighting of the silk may be obtained by including a treatment in a solution of aluminum sulfate either before or after the silicate, but this additional treatment is not commonly used at the present time.

THEORIES AS TO THE MECHANISM

Various investigators have devoted considerable time to a study of the mechanism of the tin weighting process and although it is not thoroughly understood, as stated above, certain theories have been postulated on the basis of the known facts. If boiled-off silk is steeped in a solution containing 28.73% of stannic chloride for one to two hours, it will acquire an increase in weight of from 10 to 12 per cent. It is assumed that silk has the property of forming an additive compound with stannic chloride and that this addition compound is subsequently hydrolized, thus releasing the chlorine as free hydrochloric acid. In substantiation of this theory it has recently been shown that silk which has been treated with stannic chloride and then washed, gives X-ray spectograms which are characteristic of both silk and tin oxide ($Sn O_2$) but does not give any other spectograms. If over 30% of stannic oxide is present the silk spectogram is masked.

It has also been demonstrated that although there is a definite limit to the amount of stannic chloride which a given quantity of silk will take up from one bath, this same silk may be made to take a second charge of stannic chloride by an intermediate washing and consequent hydrolysis of the first charge. In the subsequent treatments with sodium phosphate and sodium silicate the silk fiber does not play any part. The stannic acid is changed into stannic hydroxide in an alkaline solution and this compound enters into a reaction with the sodium phosphate and the sodium silicate. The exact composition of the resulting tin silico-phosphate has never been definitely determined.

DETAILS OF THE GENERAL PROCEDURE

In actual practice the sequence of steps by which raw silk is converted into tin weighted silk have been pretty definitely established. They are here listed in tabular form in order to supplement the brief outline which was given above.

1. Removal of the silk gum by immersion in a boiling soap bath.
2. Washing in a warm dilute alkaline solution containing either ammonia or sodium carbonate.
3. Washing in warm water.
4. Immersion in a cold dilute acid solution, preferably hydrochloric acid.
5. Washing in cold water.

6. Removal of the excess water in a centrifuge.
7. Immersion in a solution of stannic chloride whose gravity is not over 30° Bé. and whose temperature is about 60° F. (15° C.).
8. Removing the surplus tin solution in a centrifuge.
9. Washing very thoroughly with cold water in order to completely hydrolize the stannic chloride which is fixed on the fiber.
10. Immersion in a solution of disodium phosphate whose gravity is not over 6° Bé. and whose temperature is about 140° F. (60° C.).
11. Washing in cold water.

(Note: The steps from 4 to 11 may be repeated up to four or five times as desired.)

12. Immersion in a solution of sodium silicate whose gravity is not over 5° Bé. and whose temperature is about 140° F. (60° C.).
13. Washing in cold water.
14. Transferring to the dye bath.

Application to Silk Skeins

In the earlier days silk was always weighted while still in the form of skeins. Therefore it is fitting that our attention should first be directed to this type of weighting. The equipment in the dyehouses of those times was very meager, being usually limited to the simple open wooden vats or boxes of rectangular shape—rather narrow and varying in length as desired. The silk skeins were hung over wooden rods and suspended in the various solutions, being turned by hand from time to time in order to insure even penetration. All of the steps listed above were carried out in these open vats, the silk being lifted out and piled on wooden-slatted trays something like stretchers while on solution was drawn out and another put in. Also when it was necessary to remove the excess solutions from the skeins they were hung over a peg in the wall and wrung as much as possible through the other end of the skein.

From time to time there have been developed various improved methods for handling the skeins in the different operations so that much of the slow and costly hand work has been done away with. At present after boiling out the gum and washing, the yarn is piled into bundles and wrapped with cheese-cloth or some similar cotton fabric. The bundles of silk are placed in a special rubber covered centrifugal which is designed to operate either at a low or a high speed. A typical centrifugal of this type is shown in Plate 1. The tin solution is pumped into this centrifugal and it is run at a low speed while the silk is taking up the required amount of tin. The tin solution is then drained off and the centrifugal is revolved at a high speed to remove the excess solution from the silk. The surplus tin solution is always returned to the standing bath.

The skeins are removed from the bundles and transferred to some device for washing them with cold running water. One of the best machines for this purpose consists of a series of horizontal revolving porcelain spindles which are suspended over a box or trough in which the wash water may be collected. Just below each spindle is a horizontal perforated water pipe. The silk skeins are hung over the spindles and while they are being slowly revolved, the water is sprayed directly into the interior of the skein by means of the perforated pipe. The first wash waters after the tin treatment are run into a storage tank and saved in order to recover the tin.

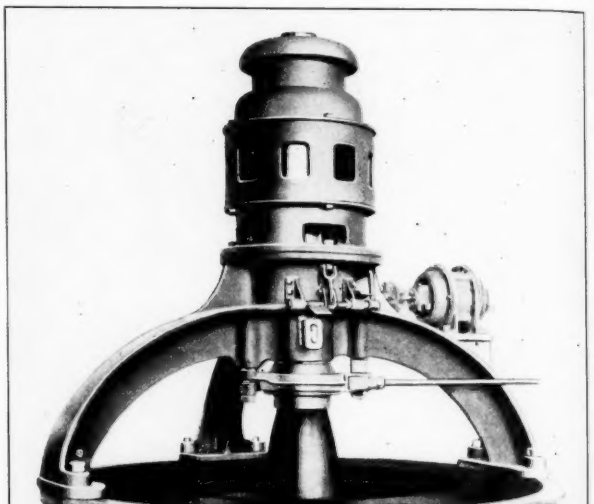


Plate 1

Combination centrifugal for weighting and extracting, with one slow speed motor for weighting and one high speed motor for extracting surplus solution.

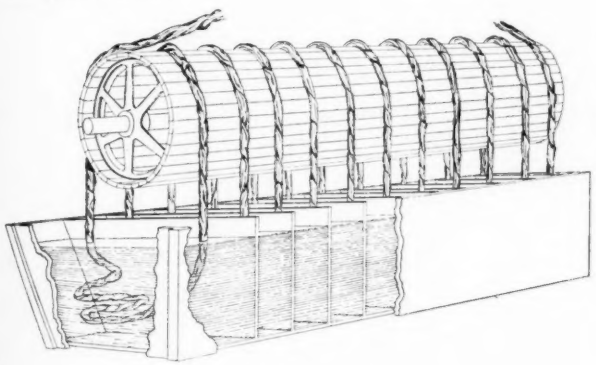
After the tin wash the silk is again bundled up and returned to the centrifuge where it is treated with the phosphate solution in the same manner as the tin. When the excess of the phosphate solution is removed, the skeins are taken to the washing machine which is described above, and the remainder of the free phosphate is washed out with running water. After the requisite number of repetitions of the tin and phosphate treatments the skeins are finally treated with silicate of soda. This treatment is still commonly carried out in the open wooden dye boxes because of the detrimental effect of the silicate solution on the centrifugal or any other mechanical equipment.

Application to Silk Piece Goods

The application of the tin-phosphate-silicate method of weighting to silk piece goods is of comparatively recent origin. It was not practiced on a commercial scale much before the year 1910 and in fact did not attain

any great prominence until more than ten years later. The first silk pieces were weighted in a manner very similar to the skeins. For example, the pieces were treated with tin either by immersion in an open box or they were wrapped in bundles and placed in the special rubber covered centrifugal. (See Plate 1.) After the impregnation with the tin solution and the subsequent removal of the excess solution the bundles were opened up and the pieces were washed either in the rope form on an ordinary overhead reel dye bark or in the full width form by passing between a succession of perforated water pipes which sprayed continuous streams of fresh water through the pieces. The treatment with phosphate could also be carried out in the special centrifugal, but it was generally performed in the regular overhead-reel bark. The wash after phosphating, the treatment with silicate and the wash after silicating were all originally carried out in these overhead-reel barks, the silk being wound up on the reel while the solutions were changed.

Considerable difficulty was experienced in obtaining satisfactory evenness by this method of weighting and this accounts for the comparatively slow increase in production during the early stages of the piece weighting game. It was found that any unevenness in weighting would result later in streaky dyeing and unfortunately it was practically impossible to tell whether a piece was unevenly weighted until it came out of the dyebath and exhibited an inferior quality of dyeing. After considerable study it was decided that a large part of the uneven weighting of piece goods was due to imperfect washing, particularly after the tin treatment where complete hydrolysis of the stannic chloride is absolutely essential. So the attention of the textile engineers was directed to the development of a satisfactory machine for washing the silk piece goods.

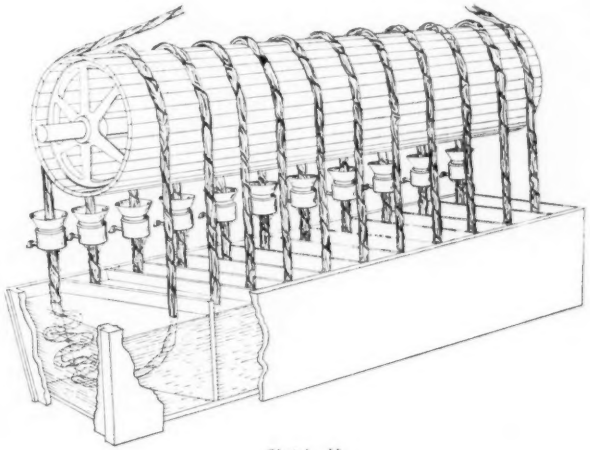


Sketch I

Single reel rope washer—first type.

Single Reel Rope Machines

One of the first types of washing machines to be developed for this purpose was known as the Single Reel Rope Washer. This machine was made with a wooden tub approximately 11 feet wide and 4 feet long with wooden framework to support a round reel approximately 27 inches in diameter and mounted about 8 feet above the tub. The tub was divided into 11 individual compartments. each compartment having an individual water overflow basin at one end. The first and by far the simplest modification of this type of washer is demonstrated diagramatically in Sketch 1. Later modifications of the same general type of machine are shown in Sketch 2 and Plate 2. In the later washers there were the same number of compartments as in the first type, but above each compartment there were spray nozzles mounted close to the round reel, in direct line with the outside diameter of the reel. The goods traveled in rope form over the reel and through the nozzle, receiving a full spray of water at this point, then into the water in the tub. After carrying along the length of the compartment the piece passed up through another nozzle or eyelet to the reel



Sketch II

Single reel rope washer—second type.

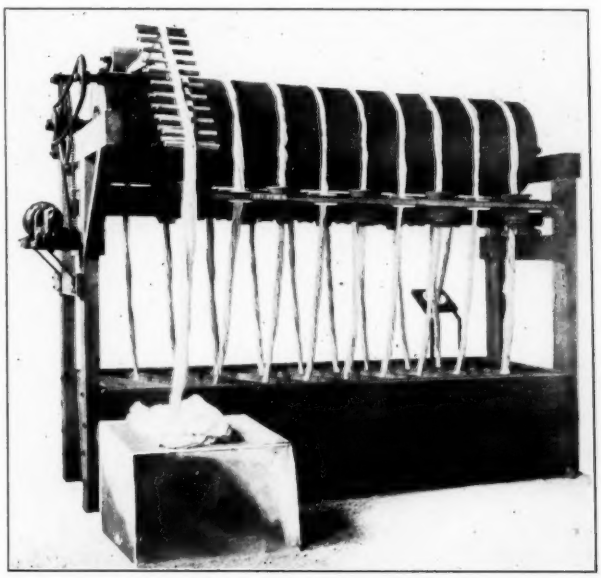


Plate 2

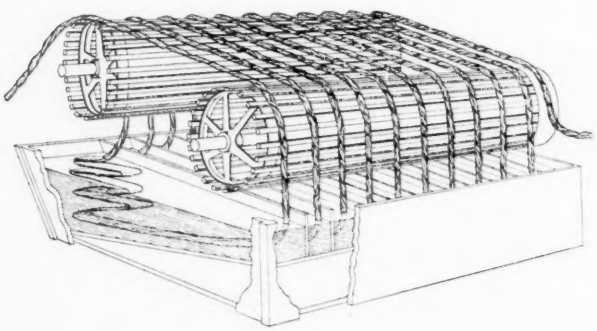
Single reel rope washer—second type.

again and so on until it progressed from one end of the reel to the other through all the compartments. The goods were then considered to have been washed sufficiently and ready to receive the phosphate treatment. This machine operated at approximately 20 yards per minute, but was subject to frequent stoppages because of "kinks" in the goods. Other disadvantages were (1) excessive pull on the goods because of the extreme height of the reel, (2) insufficient travel of the goods in the water because of the shortness of the tub and (3) excessive consumption of water.

Several modifications of this Single Reel Washing Machine were made in an attempt to remove some of the difficulties which were mentioned above. The reel was constructed with a larger diameter, the length of the tub was increased from 4 to 6 feet and the height of the reel above the tub was lessened considerably. This enabled the speed to be raised from 20 to about 38 yards per minute, but the production was still far from being uniformly satisfactory.

Double Reel Rope Machines

In 1926 a much larger machine was designed and put in operation. This machine, which was known as the Double Reel Rope Washer, is illustrated in Plate 3 and shown diagramatically in Sketch 3. It consisted of a tub 15 feet wide and 10 feet long, having 15 individual compartments with angular partitions and an individual water overflow for each compartment. The earlier models of this machine were equipped with two round reels and 15



Sketch III

Double round wheel rope washer.

nozzles, but subsequently an oval reel was substituted for one of the round reels and the nozzles were done away with entirely. The oval reel has the valuable asset of "pleating" the goods as they drop into the tub and for this reason it will "put up" more goods in a compartment than any other known type of reel. In these double reel machines the reels were built low in order to reduce the pull on the goods and the greater length of the compartments enabled the goods to have a longer flow or swim while traveling from one reel to the other through the water under the reels. It has been demonstrated that either washing, phosphating or silicating is done most efficiently in the tub and that the spray nozzle is not essential for a thorough washing of the goods. Because of the many disadvantages of these nozzles they are gradually becoming obsolete. The improved models of the

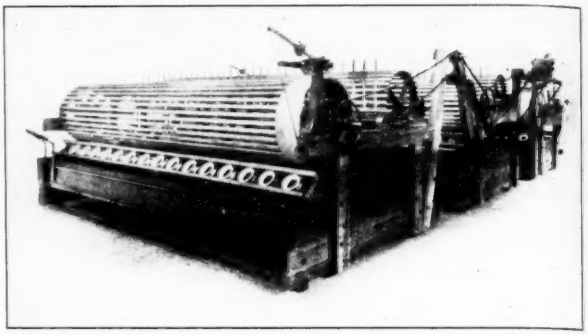


Plate 3

Double round wheel rope washer—tin washing, phosphating, and phosphate washing units.

double reel machine attained a speed of 60 yards per minute which represented a considerable increase over the best production of the single reel machines, but even yet the ideal in quantity and quality of production was not reached.

Full Width Machines

At this point some mention must be made of the attempts to build a machine which would keep the goods in the full width instead of in the rope form during the tin washing, phosphating and phosphate washing. Such a machine usually consisted of a long series of tubs or boxes in a line one after the other with a certain number of rolls at the bottom of each tub and a similar number of rolls above each tub. The revolutions of all of the rolls were synchronized so that they turned at exactly the same speed, and the silk pieces passed over the upper rolls and under the lower rolls, running into and out of the liquid in the tub which might be water or phosphate solution as the occasion demanded. Perforated pipes were usually placed under the rolls above the boxes where the goods were to be washed. The water spraying out from these pipes through the goods greatly improved the efficiency of the washing. This open width machine had the advantage of eliminating the streaks which were very apt to occur in goods which had been washed in the rope form. However, there were a number of disadvantages which more than offset this advantage, namely: (1) the goods were always under a certain tension and it was quite difficult to keep this tension constant; (2) the goods were not given long enough time in the phosphate, and (3) as a result of these factors an insufficient amount of weighting was usually obtained. Although considerable amount of experimenting has been done on this type of machine it has never proven to be very successful.

Semi-open Width Machines

We now come to a consideration of the latest and most promising development in the field of silk piece goods weighting; namely, a machine which handles the pieces in

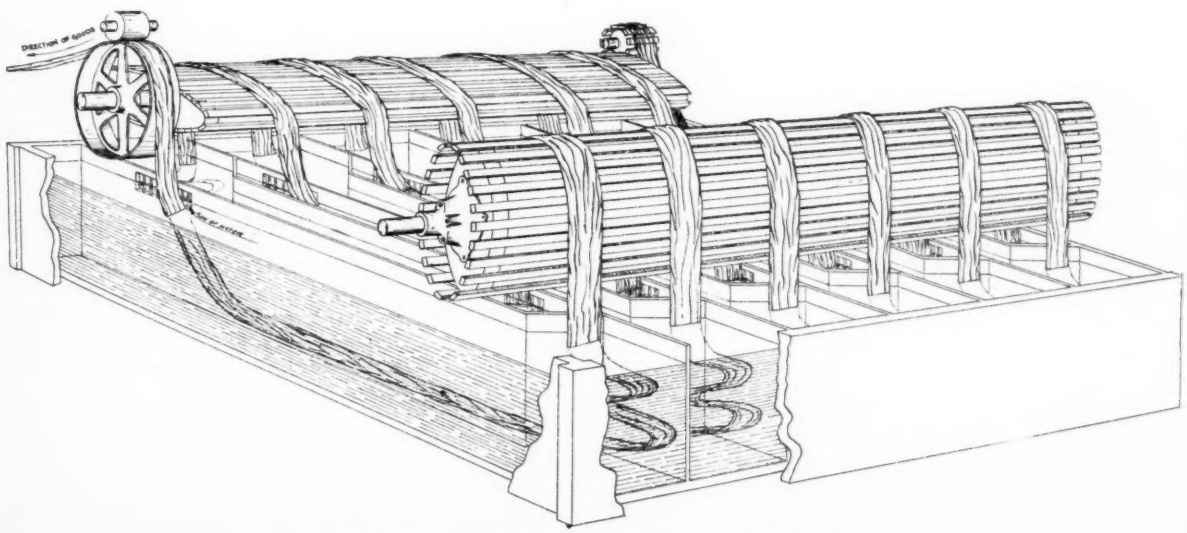
a semi-open width instead of in rope form, and carries them continuously through the tin washing, phosphating and phosphate washing processes. This machine is designed to utilize the principle of counter-current flow which is so commonly used in chemical plants for the washing of gases and other similar operations. Sketch 4 is a diagramatic representation of one unit. It consists essentially of a Cypress tub with 15 to 18 compartments (depending upon the requirements of the particular operation in question) over which is mounted on an iron framework two oval reels at suitable distances from each end of the tub. These reels are each driven by direct motor drive and they rotate in opposite directions from each other.

The goods enter the machine by passing over a primary round reel which delivers them into compartment No. 1 in loose form. Approximately 60 yards of goods are allowed to accumulate in this and in all succeeding compartments. The water or solution in the compartment is flowing in the direction to be taken by the goods so that the goods move through the compartment with the stream. They are then picked up by the oval reel at the other end and pleated into compartment No. 2. At this end of the compartment an extra large space is provided so that a considerable yardage can pile up. The piling is so designed that after the pile reaches a certain height it will fall over and automatically release the pleats. The goods then travel through compartment No. 2, again in the same direction as the current, until they are picked up by the second oval reel and pleated into compartment No. 3. The same operation of piling up and releasing is gone through again and the goods pass in a similar manner through all the compartments of the machine.

If the operation is one of washing the clear water enters the last compartment to be traversed by the goods, through a protected opening at the bottom of the partition at one end, and flows with the goods to the other end. Here it enters the next compartment through an overflow near the top of the partition and again travels with the goods through this compartment. This system of overflow at one end and underflow at the other end is repeated through all the compartments of the machine. It is known as the under and overflow circulatory gravity system and it utilizes the water to the best possible advantage. The unwashed goods enter the wash water which contains the highest concentration of washings and as the goods become cleaner so does the water, until the clean goods pass through the clear water in the last compartment. The pieces then pass between rubber squeeze rolls to remove the surplus water after which they are ready for the next operation.

The same system is utilized in treating with phosphate and with silicate. The goods enter one end of the machine and the fresh phosphate or silicate solution enters the other end. As the material passes from one compartment to the next the solution becomes increasingly stronger until in the final compartment the goods encounter the fresh solution. Just as in washing, the goods in all the compartments move with the flow of the solution. After leaving the final compartment the pieces pass through rubber squeeze rolls which squeeze out the excess of the treating solution. This excess solution is returned to the bath.

This modern machine for the treatment of silk piece goods has many advantages over the earlier machines which were devised for this purpose. In the first place it effects a great saving in water while at the same time increasing the efficiency of the washing process. Whereas in the older types of washing machines it was necessary to furnish fresh water to each individual compartment, the new principle of counter-current flow requires only one inlet of fresh water for a whole series of compartments.



Sketch IV. G. L. type square formation washing unit—latest type.

A saving in chemicals is also made possible by this machine. For example, the standing bath of sodium phosphate can be used for months by adding a concentrated stock solution to the bath intermittently, or by using a Gravity Recorder Controller to admit continuously sufficient of the stock solution to maintain the bath at a definite Baumé strength. The phosphate solution is kept circulating by means of a centrifugal pump and the fresh supplies of phosphate are introduced during the passage from the exit of the phosphating machine back to the entrance at the other end. The standing bath of sodium silicate is subject to catalytic action and therefore requires a more frequent complete change of the bath. However, a special back flow system incorporated in the machine segregates the silicate in the first four compartments where the catalytic action is greatest. The standing bath in these first four compartments is changed very often while the second standing bath in the remaining compartments of the machine may be kept for a longer period. The silicate solutions are circulated and maintained at a constant strength in the same manner as the phosphate solution.

Another advantage of this machine is that the goods are kept in a loose form in semi-open width and are under a minimum of tension. The unevenness and other difficulties due to the twisting of the pieces into a tight rope are thus eliminated.

A very important feature of the new machine, particularly from the standpoint of cost of operation, is the increased speed of production coupled with an actual decrease in labor because of lessening the need for supervision. The machine is rated to run at a speed of 95 yards per minute and it has actually been operated at a speed of 90 to 92 yards per minute without serious trouble.

Summary of Modern Piece Weighting Procedure

Before leaving this subject it seems advisable to give a brief summary of the procedure for weighting silk piece goods as it is carried out in the most up-to-date dyehouses with the aid of the latest improved types of machines.

After the boil-off the goods are properly washed and acidified and they are then wrapped in bundles and placed in the rubber covered centrifugal where the excess of water is removed by whizzing. The centrifugal is then filled with the tin solution and revolved slowly until the silk is thoroughly saturated. The temperature of the tin bath is best kept at 60° F. (15° C.). The tin solution is drained off and the centrifugal is speeded up to remove the excess solution. As these centrifugals are quite expensive the treatment with tin is sometimes given in an open wooden bark. In this case the bundles of silk are placed in baskets which are submerged in the tin bath for the required length of time. When the saturation is complete the bundles are transferred to the centrifugal for the removal of the excess tin solution.

After whizzing the bundles are opened up and the silk pieces are fed into the preliminary open washer in a continuous stream. Here the silk is kept at its full width and is passed up and down between sprays of running water. A large proportion of the hydrolysis of stannic chloride is carried out in this open washer. The goods are run directly from the open washer into the first compartment of the counter-current tin washing machine where they assume the loose, semi-open width form as described above. The unbroken string of pieces then passes through all the compartments of the tin washing unit and into the phosphating unit. The phosphate solution is best kept at a temperature of 140° F. (60° C.). The complete passage through all the compartments of the phosphating unit is so timed that the silk remains for about one-half hour in the phosphate solution. This time has been found to be amply sufficient for the complete conversion of the stannic hydroxide into stannic phosphate. After leaving the phosphating unit the silk passes on through all the compartments of the phosphate washing unit.

If the goods are to pass through another round of tin and phosphate they are again bundled up after leaving the phosphate washing machine. On the other hand, if they have already received the required number of repetitions of tin and phosphate, they are transferred directly in the semi-open width form to the silicating unit. The temperature of the silicate bath is best kept at 140° F. (60° C.). The goods pass in succession through all the compartments containing the silicate solution and then through all the compartments of the silicate washing unit. After washing out the surplus silicate the pieces are finally bundled up and whizzed in the centrifugal to remove the surplus wash water. They are then ready for dyeing.

Tin Recovery

Most of the modern dyehouses make provision for the recovery of tin from the tin wash liquors. In the typical piece weighting process described above, it is customary to save the wash waters from the preliminary open washer as well as from the first three or four compartments of the main tin washing machine. These wash waters are led into some form of settling tanks and there allowed to stand for some time. This stannic hydroxide or oxide which was formed by the hydrolysis of the stannic chloride settles down in the form of a sludge and the clear wash water can be drawn off from the top of the tank.

After a reasonable amount of tin sludge has collected in the tank, the supernatant liquor is drawn off as completely as possible and the sludge is freed from the excess of solution in a filter press. The dewatered sludge may be sold as much on the basis of the percentage of tin which it contains or it may be dried and sold in the dry form also on the basis of its tin content. In either case it is shipped to a special refinery which is equipped to smelt the stannic oxide and reduce it to metallic tin again.

(To be continued)

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HOOVER'S UNEMPLOYMENT ORGANIZATION

PRESIDENT HOOVER has called upon the business leaders of the United States to aid in the mobilization of the nation's relief agencies to meet the unemployment situation which is expected during the coming winter. Insofar as it goes, this is an important step forward in a program to eliminate the distress arising from a lack of employment.

Walter S. Gifford, director of the President's organization on unemployment relief, recently made a statement to the following effect:

"The foundation of the relief of distress, arising out of unemployment, must be complete, effective, and energetic local voluntary organization coordinating with local authorities. . . . These organizations have most successfully carried out the burden of the past winter. They are already developing plans and methods for the future. Where such voluntary organizations have not been created they should be set up at once in every city and town where there is a problem. I appeal to the business and welfare agencies to see to the creation of such definite organizations to meet the situation where such do not already exist."

Judging from the above statement, the committee is planning to have all relief measures originate in the individual localities. In other words, each community must care for its own unemployed. This is going to be a hardship on some cities and towns that have already been especially hard hit. Take for instance, a small New England town that has a population of about 2,500 people. In boom times the chief industry in that town employed 700 people on full time. At present there are about 75 people employed in the same mill and working only three or four days a week. Quite a problem for that little community to cope with.

Another situation is that where people have left one town for another. They may or may not be employed in the place to which they moved. Now the administrative forces in their new home community will have to care for them if they are not working.

In neither of the above cases would it be fair or just for the entire burden of support to rest on that particular town. In the one case the town has more than it can handle to care for so many workers—in the second case the local government organization is called upon to support families who have only been living there for a short time and have contributed little if anything to the welfare of the town in the form of taxes. In some cases they have moved from one state to another. Should the second state make unemployment adjustments for people who have spent their whole lives in some other state? Unemployment *is* a national problem and must be handled as one.

Each town, city, county, and state has contributed to the support of the Federal government and they are now entitled to Federal aid. Many localities have been able to support only one industry and when that closes down or curtails production, the inhabitants are out of a job. Diversified industries are probably a remedy for this condition but we have to contend with the present situation. On the other hand, how many different industries can a town of 2,500 people support?

This is no time to tell each local organization that they must provide their own relief measures. Too many of them are not able to do so. Every town and city would be willing to cooperate, to their fullest extent, with the state and Federal governments, but there is a limit to the extent to which they can help themselves.

DEFENSE AGAINST DEPRESSION

AT the beginning of the present economic slump, one company, a manufacturer of automatic regulating and control equipment, mapped out the following sales attack as a defense against depression:

1. Increasing the amount of advertising.
2. Paying more attention to layout and copy.
3. New products and new markets.
4. Improving older products.
5. Development and research work.
6. Strengthening organization.
7. Improving catalogue and literature.
8. Tripling amount expended at exhibitions and shows.
9. Clever sales control and direction.
10. Regular use of dramatic sales approach.

And "believe it or not" the 1930 sales of that company exceeded any previous year. By looking over the above list it is apparent that a great deal of the effort was concentrated in getting the name of the company and its products before the name of prospective purchases. Practically all of the ten items mentioned are applications of advertising in one form or another.

This company certainly took advantage of the opportunities offered during a slack period. Their prospects were in a position to be more duly impressed by publicity measures as they had more time to spend in this direction. A concern which is thus able to increase its sales during a period of depression will be in a position to grab a lion's share of the market when business conditions improve and there is an increased demand for all types of merchandise.

They have found that the introduction of new products has increased their prestige among the trade and has made their sales representatives welcome among the technical purchasing agents. Purchasing agents are inclined to give more time to new products and new processes in an effort to effect economies in their own business. It is at this time that they are more conscious of values which are presented to them through advertising. Although there may be an excess of old products on the market the new ones are able to find a place and bring up the sales average for a given period.

Although the same method of attack as here outlined has proven successful with one company it cannot be taken as granted that the same plan will prove successful with some other concern. Each organization must analyze its own production, sales, and market and then map out its individual strategy.

TEXTILE CHEMICALS IN THE UNITED KINGDOM

According to Vice Consul Wallace E. Moessner of Manchester in a special circular of the Department of Commerce, the United Kingdom is one of the leading countries of the world as a producer, consumer, importer, and exporter of the chemical products and dyestuffs used by the several branches of the textile industry.

To indicate the extent of the home demand for these chemicals, it is estimated that approximately 4,175,000,000 square yards of cotton, 480,000,000 square yards of rayon and rayon mixtures, 350,000,000 square yards of woolen and worsted, 170,000,000 square yards of linen, and 15,000,000 square yards of silk piece goods as well as other textile manufactures, are each year bleached, dyed, printed or otherwise finished within the country. In addition, about 40,000,000 pounds of rayon yarn are produced by the use of chemicals.

The present total annual consumption of acetic acid amounts to about 15,000 tons (1 ton = 2,240 pounds); bleaching materials, 85,000 tons; sodium compounds of all kinds, 1,000,000 tons; finished coal tar dyes, 16,000 tons; and natural dyes and dyewood extracts, 2,800 tons. Considerable quantities of each of these products are, of course, used for purposes other than textile manufacturing and finishing, but it is not possible to determine the quantities consumed for the various purposes.

In the acid group the chief products used by the British textile industry for bleaching, printing, finishing, and similar processes, include acetic, hydrochloric, sulphuric and tartaric acids. Other products consumed in considerable volume are alum and aluminous derivatives, ammonia and ammonium compounds, barium derivatives, borax, calcium compounds, chloride of lime, chromium compounds, certain copper derivatives, Glauber salts, hydrogen peroxide, magnesium chloride, magnesium sulphate, niter cake, potassium compounds, soda ash, caustic soda and other sodium compounds, tin salts and certain zinc compounds. For sizing and stiffening, glue, gelatine, starch dextrine, sago, resins, china clay, chalk, etc.

The natural dyes used most extensively include those obtained from cutch, annatto, cochineal, cudbear, fustic, indigo, logwood, madder, orchil, Persian berries, and quercitron. In synthetic coal tar dyes, the principal demand is for blue vat colors, red chrome and mordant, direct cotton black, acid wool black and sulphur black.

The prosperity of chemical and dyestuffs manufacturers is largely dependent on the economic position of the industries consuming their products. Although faced with a declining demand in the home market textile chemical manufacturers have succeeded in obtaining a larger share of the existing business and have consequently been able to maintain and in some cases materially increase their volume of output. Furthermore, as the result of large mergers, greater research, and more modern methods of manufacture, production costs have been lowered, price agreements have been entered into and more efficient sales methods arranged, all of which has tended to maintain the economic position of the industry on a favorable level as compared with other basic industries. Perhaps the most notable example is the rapid development and expansion of the dyestuffs industry during the past decade under the protection of the Dyestuffs Act.

During the last year the home trade in alkalis and products was restricted on account of the decreased demand from rayon makers. In the case of chlorine products there was only a slight decline while severe competition was encountered in bleaching powder, this material being displaced by bleaching liquors made in consumers works from chlorine. The decline in the output of cellulose acetate rayon was also partly responsible for the decline in domestic sales.

A new plant for producing liquid chlorine was recently put into operation by the Staveley Coal and Iron Company, and Imperial Chemical Industries have concentrated the manufacture of soda crystals by removing their electrolytic caustic and chlorine plants from Gateshead to Billingham.

A few years ago the United Kingdom was almost wholly dependent on imports for its supply of acetic acid, while at present the product is being manufactured by Imperial Chemical Industries and by the Distillers Co., using the acetaldehyde oxidation process. Despite the depression in the rayon industry, the consumption of acetic acid has not appreciably diminished.

A list of manufacturers of textile chemicals and associations purchasing textile chemicals for a large part of the textile mills may be obtained on application by properly accredited United States firms, from the Chemical Division.

Commercial Grading of Sulfonated Oils

By **RALPH HART**
Hart Products Co.

This article resulted from a request made of the Bureau of Standards in Washington by one of the countries largest users of sulphonated oils that they cooperate in the establishment of commercial standards covering grading methods for this commodity. The author was called upon to assist and his suggestions are embodied in the following article. —*Editor.*

SULFONATED oils are practically always graded on a percentage basis, though the exact constituent in the oil to which the percentage refers is a matter of dispute. According to one classification it represents the combustible fatty matter; according to another it refers to the same ingredient plus any inorganic constituents which are chemically combined with the oil; whereas according to a third, the percentage stands for the difference between 100 per cent and the per cent moisture.

The first classification or "fat-methods" is still further complicated in that there is no agreement on the definition of the fatty matter. It may refer to the original raw oil, in which case the percentage actually expresses the yield obtained during manufacture. Or it may represent the fat obtained upon analysis by decomposition with acids. This analytical method is likewise unsatisfactory because of the many volumetric and gravimetric methods employed, with considerable divergence in the results. By still another fat-method, the percentage represents total combustible fatty matter as it actually exists in the finished oil, and is obtained indirectly "by difference." Furthermore, nearly all of the fat-methods are objectionable to the oil manufacturer, since the results are usually lower than by the other methods and apparently would show up his product to disadvantage.

The second classification mentioned above, in which the combined inorganic constituent are included with the fatty matter, seems to be a reasonable grading. It is objectionable, however, from the point of view that a very complete analysis is required in order to arrive at the result; another objection is the fact that detailed procedures for the individual determinations have not been standardized. The third classification, namely the "water-method," is the simplest from an analytical standpoint and is the one most commonly used. It yields somewhat higher results than the second classification as the non-essential inorganic matter, always present more or less in all sulfonated oils, is included. The difference in the grading of the same oils by some of the methods in use is shown in Table I.

With such confusion it is not surprising that a large consumer of sulfonated oils has appealed to the Bureau of Standards at Washington "for cooperation in the establishment of a Commercial Standard covering test methods for this commodity." Mr. L. B. Gilbert of the Division of Trade Standards, Bureau of Standards, has discussed the matter with the writer with a view of complying with this request. The following suggestions are offered:

It is conceded that a satisfactory commercial standard should be rational and definite, fair to both consumer and producer, and easily checked. The writer believes that the "water-method," with proper modifications, comes the nearest of meeting these requirements. The water may be determined either by volatilization or directly by distillation with xylol. It is the most definite and probably the simplest item in a sulfonated oil to determine, and with the proper precautions concordant results by different analysts may be expected. For this purpose, pure sulfonated oils may be considered to consist of the following:

A Essential ingredients—fatty matter, true sulfonated oil and soap.
B Non-essential non-volatile ingredients—salts and other impurities.
C Water.

Grading by the water-method consists simply of determining the per cent moisture and subtracting the result from a hundred. Evidently the oil manufacturer is given credit for the organically combined inorganic constituents, as it is included with the fatty matter. This is reasonable since it costs as much, if not more so, to introduce these elements in the oil as their equivalent of fat. Moreover these ingredients are vital since the character and value of the product depend upon the amount and the skill used during manufacture in combining them chemically with the oil. It may be necessary, however, to limit the amount of alkali to be included with the fatty matter.

The water-method furthermore is also applicable to those oils now on the market which are truly sulfonated, i.e., they do not split off their combined SO_3 upon boiling

TABLE I

	Sample No. 1	Sample No. 2
	Per cent	Per cent
Fat-method by difference..	64.2	67.0
Fatty matter plus combined inorganic	72.7	73.7
Water-method	73.5	75.7

with acids. Objections may be raised against including with the fatty matter the non-essential inorganic ingredients. This criticism, however, is not entirely justified as the following consideration will show. Sulfonated oil as is well known is made by treating raw oil with concentrated sulfuric acid and removing excess of the latter by washing with salt solution. No matter how carefully made or how long the oil is settled, there is bound to be some salt left in the oil—to be sure very little in a good oil. Within certain limits therefore a small amount of salts is not an adulteration and is beyond the control of the manufacturer. Hence the modification suggested to the water-method is that the amount of impurities be disregarded, provided it is within certain limits.

In including thus the non-essential ingredient in the result, we have a precedent in oil analysis. Practically every natural saponifiable fat contains a certain, small amount of unsaponifiable; yet if the latter is within specified limits it is disregarded and the oil is considered pure.

Evidently a method is also required for determining the inorganic constituents which need be used only to the same extent as unsaponifiable in oils. There are two current methods available; (a) by washing the oil, dissolved in proper solvents, with ordinary salt solution and determining the sulfate in the wash-water or (b) by the writer's indirect method from the ash, organically combined SO_3 and fixed alkalinity as soap.

Referring to the writer's method for the complete analysis of sulfonated oils published in this Journal (April 27th and May 11th, 1931) he would make the following additions in order to make the method more rigid: After making the SO_3 determination by titrating the decomposed mixture, the latter is again acidified, the fat extracted with ether and washed free from mineral acids. The saponification values of the extracted fat and of the original sample are now made. The saponification value of the former based on the sample taken for analysis should agree with the saponification value of the original sample plus the fixed alkalinity corresponding to soap. If they do not, the sample is not a pure oil and special methods of analysis are required. Evidently the unsaponifiable may be determined in either sample after the saponification test, preferably the extracted fat sample.

A New Method for the Determination of Inorganic Impurities in Sulfonated Oils

By Ralph Hart
Hart Products Co.

In A previous paper (this Journal April 27, 1931) it was proposed that sulfonated oils be graded according to their content of fatty matter plus the organically combined inorganic constituents as they actually exist in the oils. To that end it is necessary to determine the moisture and the amount of inorganic impurities in the oil. As pointed out elsewhere, no simple direct method for inorganic impurities in sulfonated oils is available or has thus far been described. A method is herewith suggested which is based on the finding of matter in soap insoluble in alcohol. Instead of alcohol, however, xylol is used as the solvent and furthermore the solution before filtering is made anhydrous by boiling.

A preliminary investigation indicated that the direct method is capable of yielding quantitative results. The following procedure was employed: The test for inorganic impurities was combined with the determination of water by the xylol-distillation method, using a reflux condenser. After all the water was expelled, oleic acid about twice the weight of the sample taken for analysis, was added to the solution and filtered boiling-hot on a counterpoised filter paper. The residue was then washed three times with 20 cc. portions of hot xylol, twice with 20 cc. each hot absolute alcohol, and twice with 20 cc. portions of ethyl ether. The filter paper was finally dried to constant weight. A Gooch crucible with suction may be used instead of the filter paper. As a precaution against the presence of organic matter due to incomplete washing, the residue may be ignited and weighted.

Oils were analyzed in this manner to which were added several per cent of sodium sulfate. Tests for sulfates were made on the filtrate by shaking with sodium chloride solution and testing with barium chloride; the results were invariably negative. Moreover the loss upon igniting the dry residue was practically negligible, indicating that the oils and soaps were completely removed by the procedure for washing as outlined. The purpose of the oleic acid is to keep the soap-oil-xylol mixture from jellying upon cooling. There is also a tendency for the sodium sulfate in the oil when boiled with xylol to be thrown out in a gelatinous form, greatly interfering with the filtering. This difficulty, however, is readily overcome by filtering boiling-hot, or by the use of a Gooch crucible with suction.

The writer is greatly indebted to Hugh H. Mosher, chief chemist for the U. S. Testing Laboratories, New York, for several valuable suggestions.

New Bulletins

The Roots-Connersville-Wilbraham Division of the International-Stacey Corporation, Connersville, Indiana, has just issued four new bulletins on gas pumps.

These bulletins supersede all former bulletins on these products issued separately in the past by The P. H. & F. M. Roots Company, The Connersville Blower Company, Inc., both of Connersville, Indiana, and the Wilbraham-Green Blower Company of Pottstown, Pa.

French Coal-Tar Production in 1930

During 1930 the French production of coal-tar showed a slight increase. The French Coal Tar Producers' and Distillers' Association represents for French and the Sarre district, 93.5 per cent of the production of coal tar and more than 99 per cent of the coal distillation.—*World Trade Notes.*

Seller Go Easy—Buyer Beware!

By D. B. Alexander

Many textile laboratories could undoubtedly recall numerous experiences similar to those noted by the author of this article. In these days when fractions of a cent have to be cut off here and there in the cost of manufacturing it would appear foolish to curtail expenses by dimissing the chemist. In many instances it would be more to the point to engage an additional one.

THE seller of any commodity has the right to charge whatever price he chooses within the bounds of law and reason and to make as large a profit as is possible. It is the business of the buyer to see to it that he gets the best possible value for his money.

Many manufacturers and dealers make large profits on the products they sell and this is permissible if the Buyer is willing to let them get away with it.

Most of our industries of a few years ago made huge profits. They formed the habit of large profits but would they have found themselves in their present precarious circumstances had they not been able to make such large profits? We often have heard the statement of a few years ago that such and such a plant was well managed where, as a matter of fact, it was inefficiently managed, but as it was able to dispose of its goods at a substantial profit, poor management did not show up. Today with decreased profits and greater competition, management must be more efficient to survive, yet it must not become Efficient Minded to such an extent that it demoralizes the help as some plants have done. Contented help means increased production and quality; discontented help may ruin the plant. Some of our so-called well managed businesses of the present day may show inefficiency as the number of their more efficient competitors increases and competition becomes keener. Moral! Become Efficient Now!

We can not compare successful industries of twenty-five years ago with the same industries of the present time although many point with pride to the past. An industry making small profits to-day is very apt to be better managed and in most cases must be better managed than the same industry years ago.

To-day to survive the struggle for existence, it becomes necessary for every industry to purchase their raw materials at the lowest possible price. Superintendents and overseers must see that the cost of their raw materials is as low as possible. This they should do not only for the sake of the management but for the laborers in the plant whose very livelihood may depend upon the plant being kept running. A few years ago the successful overseer and superintendent could purchase his raw materials at any price and was considered a success as long as he turned out good work and the plant made money. They would often be heard to say "Why use something cheaper (even if the same thing) as long as we are getting good results and having no trouble with what we are using." Today a man is not considered a success unless his work is first class and in addition must keep the cost of his raw materials as low as possible.

Many textile mills have payed and still continue to pay high prices for their raw materials. Just how much longer they can do so and continue in business is problematical. A small loss here, a small loss there, a large loss here, and a large loss there, when totaled for the entire plant may run into thousands of dollars.

Many textile mills are purchasing their raw materials from foreign countries when the same materials can be purchased at home for the same price or at a better price. It would seem to be our patriotic duty to make use of home-made materials when available and thus furnish employment to some of our Unemployed.

Many of our large cities now have "Better Business Bureaus." Why not "Better Business Bureaus" for our Textile Industries?

Many of our clients sends in samples of their purchases for test and below are some of our findings as regards Textile Chemicals offered the textile industry to-day. Losses are estimated per barrel of four hundred pounds, the loss to any particular mill depending upon it's consumption of the product.

Textile Mills have lost considerable money on the dyestuffs they have and still do purchase. We have a large number of American and Foreign Dyestuff Manufacturers who sell their products directly or indirectly to the textile mills. When sold indirectly the manufacturer sells to a Dealer or Jobber who not infrequently owns a mixing plant where he adds salt or dextrine to the dyestuff thereby reducing it's strength. The Dealer or Jobber then resells the diluted dyestuff to the textile manufacturer not infrequently at a higher price than the manufacturer would have sold the undiluted dyestuff directly. Not all Dealers or Jobbers salt this way and many are legitimate representatives of the manufacturers, but those that do are Parasites on the Textile Industry and should be Cast Off. There is no objection to purchases being made from a Dealer or Jobber when quality and price are as good as can be obtained from any other source.

In the past a good many, a great many dyers have formed the habit of buying mixtures of dyestuffs but to-day the larger dyestuff manufacturers are advocating that the dyer purchase separate (straight) dyestuffs and

do his own mixing. However, to satisfy dyers that desire mixtures, all manufacturers will sell mixtures. There are several reasons why it is better not to buy mixtures:

1. A dyer is using considerable amounts of a brown mixture and purchases two or three barrels at a time when suddenly the orders for the brown shade are cancelled and the dyer finds he is stocked with a dyestuff that he cannot use. The dyer would not have been caught in this difficulty had he purchased the ingredients of the mix as straight dyestuffs and mixed his own. The straight dyestuffs left on hand could be blended to produce new shades that might be in demand or be used as straight dyestuffs. When the dyer mixes his own a lower inventory of dyestuffs need be carried and less money tied up.

2. The mixture may contain several different dyestuffs and the dyer finds that he must use additional dyestuffs for shading. This may make it difficult for him to get his shade and especially to do repeat work for with several dyestuffs in the dyebath he is bound to find variations in the rates of exhaustion of the various dyes, some coming off at the boil, others going on and vice-versa. The fewer dyestuffs in the bath the better. If the dyer using a mixture must weigh out small quantities of shading dyestuffs he might as well weigh out straight dyestuffs at the start and not use a mixture.

3. Not infrequently a mixture is composed of an expensive dyestuff and a cheap one yet the seller will charge for the mixture the price or more than the price of the expensive component when purchased alone.

4. A good many dyestuff manufacturers charge for mixing. Why not pay one of your own men to do the work?

A particular dyestuff is often manufactured by different manufacturers and each gives the dyestuff a name with the result that the same dyestuff may have as many as twenty-five names. A number of dyestuffs have been carefully investigated and their composition, uses, fastness properties, tests for identification, patent and literature references, and trade names tabulated. The same dyestuff regardless of it's trade name is given a number known as the Color Index Number. There are some good dyestuffs on the market that have not yet been identified and given Color Index Numbers. However, the majority of colors needed for turning out good work have Color Index Numbers and it would be a good plan if the dyestuffs for the greater part of textile work were bought and sold by Color Index Number. Why not bring order out of chaos and in the majority of cases adopt Color Index Numbers as a basis for dyestuff purchase?

One large company purchased a considerable amount of Tartrazine from a dealer under the name of Fast Light Yellow 2 G and the purchase was made under the belief that they were buying a true Fast Light Yellow which is about twice as expensive a dyestuff as Tartrazine and one-half as strong tinctorially. This was a deliberate case of misrepresentation and cost the company making the purchase considerable money. Loss per 400 lb. barrel—$560.

An acid blue mixture purchased from a small manufacturer sold for $1.40 a pound and was replaced by a similar mixture purchased from another manufacturer for $1.70 but twice as strong tinctorially. Loss per 400 lb. barrel—$220.

A dealer sold a straight acid blue for $3.60 a pound. When purchased directly from the manufacturer the price was $3.00 a pound and the dyestuff twenty per cent stronger. Loss per 400 lb. barrel—$480.

A client recently purchased an acid green under a standard name used by many manufacturers to denote an unmixed dyestuff. The product was an exceptionally good purchase as far as dyestuff strength (tinctorial) was concerned and sold for $3.10 a pound. Upon anaylsis the dyestuff was found to be a mixture of two dyestuffs one ingredient costing $2.90 a pound, the other $1.75. Our client lost on a 400 lb. barrel—$120.

One concern purchased an acid blue from a jobber for $2.80 a pound and when this dyestuff was purchased directly from a manufacturer for $2.50 a pound a stronger product was obtained. Loss per 400 lb. barrel—$150.

A woolen mill using chrome colors was found to be loosing the following amounts per barrel of 400 pounds:

Chrome orange	$540
Chrome red	750
Chrome brown	500
Chrome yellow	300
Chrome black	400
Chrome blue	100
Chrome blue	100 (This dyestuff was a mixture of two 60c a pound dyestuffs and sold for 70c a pound. The mixture was also let down with salt.)

Another manufacturer using chrome colors lost the following amounts per barrel of 400 lbs.:

Chrome blue	$280
Chrome brown	240
Chrome black	160
Chrome red	300
Chromate red	800
Chromate red	452

A sulphur blue was in use and cost a company 55c a pound. This was replaced by a similar product which met all fastness requirements for 40c a pound and the new product was 15 per cent stronger. The loss on the old color per 400 lb. barrel was $84.

A sulphur brown purchased from a dealer cost 48c a pound and was replaced by a similar product of equal fastness properties and twice as strong tinctorially but costing half as much (24c). Loss per 400 lb. barrel $144.

A direct color cost one concern 60c a pound when purchased from a small manufacturer. The same type color of equal strength and fastness was purchased from another manufacturer at 35c a pound. Loss per 400 lb. barrel $100.

Throwster's Tints for silk throwing are often purchased at high prices when suitable colors are available from other sources at much lower cost. Known losses per 400 lb. barrels of Throwster's Tints have been:

Throwster's rose	$500
Throwster's green	425
Throwster's blue	300
Throwster's yellow	400
Throwster's red	450

Many starch, softener, and size dealers and manufacturers make considerable money at the expense of the Textile Industry. At the present time the management of most Textile Mills could make good use of the money thus lost.

One mill purchased British Gum at 11½c a pound when a similar product was available at 5½c a pound. Loss per 400 lb. barrel $24.

Another concern saved $3.00 a day by the selection of the proper corn dextrine to do their work.

One manufacturer paid 4.8c a pound for a 40% soluble corn dextrine while another paid 6c a pound for the same material. Loss per 400 lb. barrel $4.80.

Rayon sizes are available at all prices. One mill paid 33c a pound for rayon size that they could have duplicated for 12c a pound. Loss per 400 lb. barrel $84.

A sizing containing 96% water was sold for 7c a pound. This material could be duplicated in the plant for 2c a pound. Loss per 400 lb. barrel $20.

Another sizing compound sold for 7½c a pound and was a mixture of potato starch and Aktivin. This size could have been put together in the plant for 4c a pound. Loss per 400 lb. barrel $12.

Another sizing in use in several mills sells for 11½c a pound. This product could be duplicated for 8½c a pound. Loss per 400 lb. barrel $14.

An Olive Oil emulsion for finishing was sold for 8½c a pound and could be readily put together in the mill for 5c a pound. Loss per 400 lb. barrel $14.

Cream Softeners have cost a good many mills plenty of money in the past. A cream softener now in use by one company sells for 7½c a pound, a similar and equally good a product being available at 4½c a pound. Loss per 400 lb. barrel $12.

Another softener selling for 9c a pound can be duplicated for 6c a pound. Loss per 400 lb. barrel $12.

Soaps, Scouring Compounds, Paint and Tar Removers, Stripping Liquors, Wetting Out Agents are other materials upon which money is lost unless the products are well scrutinized and only the best purchased and at proper prices.

A commercial Wool Scouring Plant was offered a scouring liquor at 9½c a pound for which wonderful claims were made. Upon analysis the material was found to be a weak solution of caustic soda worth ½c a pound. Caustic soda could be used in Wool Scouring if properly controlled, but the hoped for buyers of this dangerous solution were not told of its composition and had the operators tripled the amount recommended for use injured wool would have resulted. Loss per 400 lb. barrel $36.

(For the benefit of the skeptical—one commercial wool scouring plant used caustic potash for several years in doing their wool scouring.)

One of the best grades of sulphonated castor oil on the market contains 65% fatty acids and sells for 12c a pound. A certain dyehouse paid 15c a pound for an inferior oil containing but 32% fatty acids. Loss per 400 lb. barrel $36.

Another dyehouse paid 11½c a pound for a sulphonated oil-pine oil mixture which could have been purchased for 5½c a pound. Loss per 400 lb. barrel $24.

A benzine-sulphonated castor oil mixture used in several plants sells for 12c a pound. A suitable substitute is available at 5c a pound. Loss per 400 lb. barrel $28.

A thin pouring scouring compound for use in a woolen mill sold for 9½c a pound. This was replaced by a similar product for 5c a pound. Loss per 400 lb. barrel $18.

Another type of scouring compound resulted in a loss per 400 lb. barrel of $14.

A Worsted Mill purchased a Tar Remover for 15c a pound. The product was a mixture of cresylic acid and sulphonated oil and could be obtained from another source for 9c a pound. Loss per 400 lb. barrel $24.

Another Tar Remover sold for 13c a pound and could be obtained for 4½ a pound. Loss per 400 lb. barrel $34.

One concern purchased a Moth Destroying Liquid for $4.00 a gallon. They could have put the mixture together themselves and saved on a 400 lb. barrel $110.

A wool cleanser sold for 7c a pound, could be purchased for 4c a pound. Loss per 400 lb. barrel $12.

We found a dyehouse using considerable amounts of Titanous Sulphate as a Stripping Liquor. They paid a high price for the product. Loss per 400 lb. barrel $20. They saved money when they replaced the Titanous with a Hydrosulphite-formaldehyde compound at our suggestion.

A dealer sold Sodium Acid Sulphate, a wool dyeing assistant, for 6c a pound. The same material could be purchased for 3c a pound. Loss per 400 lb. barrel $12.

A yarn dyehouse was using a wetting-out agent that cost 35c a pound. One pound of the product could be replaced by 4½ ounces of another well known wetting-out agent selling for the same price. Loss per 400 lb. barrel $100.

Vat Dyestuffs of the Benzanthrone Series

German Patent No. 451,122 (21/X/'27)—*I. G. Farben-Ind. A.-G.*—Reference is made to oxidation products of dibenzanthrone (G. P. No. 259,370) as the basis of the reaction covered. These products are treated with esters of strong acids with polyvalent alcohols, the acids not necessarily being halogen acids. The ester must have at least two hydroxyl groups, attached to different chain carbons, esterified. A more special claim covers polyvalent alcohols in which one or more hydroxyls are esterified by aryl-sulphonic acids. The products are blues, in the four examples given.

Effect of pH Upon The Detergent Action of Soap*

By **F. H. RHODES** *and* **C. H. BASCOM**
Cornell University, Ithaca, N. Y.

The detergent action of an approximately neutral soap, at the optimum concentration of 0.25 per cent and the optimum temperature of 40° C., at first increases and then decreases as the alkalinity of the solution is increased. The maximum detergent effect is obtained at a pH of 10.7. At this optimum alkalinity the increase in the detergent power of the soap appears to depend to a marked extent upon the nature of the anions present. In general, the effect of an anion in improving the efficiency of a soap at the point of optimum alkalinity appears to increase with increase in the valence of the anion.

IN COMMERCIAL laundry work it is common practice to add to the washing solution certain special addition agents, or builders, which increase or modify the detergent action of the soap. In some instances the effect of the builder appears to be due to its action in altering the concentration of hydrogen ions in the washing solution. In other cases the addition agent may aid in forming or in stabilizing the froth, or it may act as an adsorbent or as a solvent for some of the components of the dirt. On the basis of their modes of action, we may classify the commonly used builders as follows:

(1) Substances that owe their effects principally to their action in modifying the concentration of hydrogen ions in the washing solution (sodium hydroxide, sodium carbonate, trisodium phosphate, sodium silicate, borax).

(2) Foaming agents (ricinoleic acid, casein, etc.).

(3) Adsorbents (talc, bentonite, sodium silicate).

(4) Solvents (naphtha, tetralin, hexaline, etc.).

The present investigation was confined to the study of the effects of those addition agents that change the concentration of hydrogen ions in the soap solution and to the determination of the relationships that exist between the pH of the washing solution and its detergent action. The builders studied were sodium hydroxide, sodium carbonate, sodium borate, and trisodium phosphate. A few experiments were also made with sodium silicate. There are, however, so many different types of sodium silicate and its action is so complicated by the presence of the colloidal silicic acid that this builder is being made the subject of a separate investigation.

Previous Work

Some information as to the effects of alkaline builders upon the properties of soap solutions has already been published. Elledge and Isherwood (3) found that the addition of alkaline substances to soap solutions decreases the surface tension and increases the ability of the solutions to carry carbon black into suspension. They did not make any actual washing experiments nor did they measure quantitatively the concentration of hydrogen ions in the solutions used by them. Richardson (10) states that the surface tesion of a soap solution is decreased by the addition of sodium silicate, although the effect produced by this compound is not so great as that obtained with sodium carbonate. Millard (7) found that the silicate, phosphate, carbonate, and hydroxide of sodium all decrease the surface tension of soap solution, the effect of the silicate being the least and that of the hydroxide the greatest. All these investigators assume that the surface tension of a soap solution is, qualitatively at least, an index of the detergent power. In no case were their conclusions as to the effects of alkaline addition agents upon the detergent power of soap confirmed by actual washing experiments made under conditions approximately those of laundry practice.

Shorter (11) found that the ability of a solution of soap to stabilize a suspension of benzene in water is greatly increased by the addition of small amounts of free alkali. He concluded that the detergent power increases with the alkalinity. His conclusion is based upon the unproved assumption that the detergent power is proportional to the ability to stabilize colloidal suspensions of benzene.

Fall (4) showed that a solution of sodium silicate can retain considerable amounts of manganese dioxide in suspension, even in the absence of soap. The hydroxide, carbonate, and phosphate of sodium also possess some peptizing action within a rather narrow range of concentration. Sodium borate does not carry manganese dioxide into stable suspension. Vincent (12) found that the ability of soap solution to emulsify oils is increased by the addition of small amounts of sodium hydroxide or trisodium phosphate, but decreases again when the concentration of the added substance exceeds a certain optimum value. Neither of these investigators obtained any quantitative data as to the relationship between the concentration of hydrogen ions in the soap solution and its detergent power. Berzell (2) measured quantitatively the amount of cleansing accomplished when soiled woolen cloth was washed with soap solutions to which various amounts of alkaline builders had been added. The experimental errors inherent in his method are so large that the results

*Industrial and Engineering Chemistry.

have little quantitative significance. Mullin (8) discussed the general theory of the effect of the addition of alkaline substances upon the efficiency of soap as a cleansing agent. He gives few experimental data, but makes the general statement that, within the range studied by him (from pH 7 to 13), the cleansing power of a soap solution is more or less proportional to the pH of the solution. He further says that in wool scouring it is commonly recognized that the first small additions of alkali increase the effectiveness of the soap, but that the effect becomes progressively smaller with further additions.

Experimental Procedure

Solutions were prepared that contained a constant concentration of a neutral soap and varying amounts of alkaline salts. The pH value and the detergent power of each such solution were measured and the variation of detergent value with the change in the concentration of hydrogen ions was determined.

The standard soap used was a commercial soap flake. It showed the following analytical characteristics:

	%
Moisture	1.72
Free alkali	0.00
Free fatty acid	0.00
Salt (NaCl)	0.82
Glycerol	0.4
Total alkali (as Na_2O)	10.91
Titer of fatty acids	34.96° C.

All solutions were made up to contain 0.25 per cent by weight of dry soap, because Rhodes and Brainard (9) have shown that, with this soap, solutions of this concentration are as effective in cleansing as are more concentrated ones. The alkaline addition agents used were sodium hydroxide (containing less than 0.1 per cent of sodium carbonate), sodium carbonate, sodium borate (borax), and trisodium phosphate. All solutions were prepared with distilled water which had been boiled to expel carbon dioxide.

The concentration of hydrogen ions in each solution was determined by measuring the electromotive force developed between a standard calomel electrode and a hydrogen electrode immersed in the solution to be tested. Preliminary experiments made with buffer solutions of known pH values showed that this method and apparatus would give accurate results.

Effect of Adding Builders on pH of Soap Solution

The standard solution of soap alone had a pH of 10.16. This value is of the same general order of magnitude as the results obtained by other investigators who have measured the concentration of hydrogen ions in soap solution (1, 5, 6). Of course, an exact quantitative agreement is not to be expected, since the earlier measurements were made with different soaps at a different concentration and a different temperature.

The effects of the addition of alkaline builders upon the pH are shown in Figure 1. Sodium hydroxide, trisodium phosphate, and sodium carbonate all increased the pH of the solution—i. e., decreased the concentration of hydrogen ions—the hydroxide having the greatest effect and the carbonate the least. The relationship between the pH

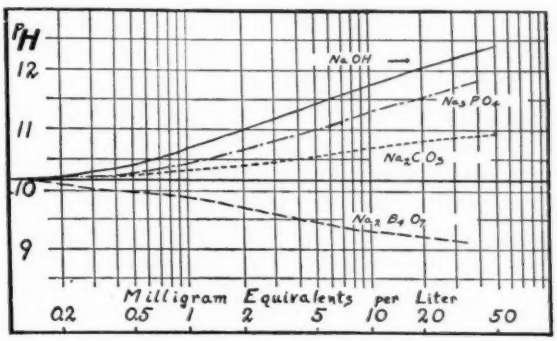

Fig. 1

Effect of addition of alkaline builders on pH of solution.

and the logarithm of the molar concentration of the builder was approximately linear throughout the range of concentrations studied. With sodium borate the pH decreased more or less regularly with increase in concentration of the builder. This is to be expected, since the borate itself is less alkaline than the soap.

Detergent Effect of Builders

The detergent powers of the various solutions were measured quantitatively by the method described by Rhodes and Brainard (9). Standard soiled cloth was washed under controlled conditions with a fixed quantity of a solution of known concentration of soap and builder, and the brightness of the washed cloth was measured by means of a reflectometer. The washing was repeated with fresh portions of the cleansing solution until the cloth had been washed five times. The conditions during washing approximated those of ordinary laundry practice. This method measures the detergent power of the cleansing solution directly.

The standard cloth used was a cotton muslin with a thread count of 58-68. The cloth as purchased contained starch sizing which interfered with uniform soiling and washing. To remove the starch, strips of the cloth 46 cm. long and 10 cm. wide were immersed in a solution containing 3 grams of commercial malt diastase in 1 liter of distilled water. After the cloth had soaked for 3 hours at 48-50° C., 1 liter of distilled water was added and the cloth was boiled in the diluted solution for 10 minutes. The muslin was then rinsed twice in distilled water, boiled for 10 minutes in a solution containing 0.5 per cent of soap, rinsed thoroughly with several portions of distilled water, wrung dry, pressed flat between several layers of cheesecloth, and dried in the air.

The procedure followed in soiling the cloth, washing, and measuring the brightness of the washed cloth was identical with that described by Rhodes and Brainard (9). All washings were made at 40° C., since it has been shown

that the detergent action of the particular soap used in these experiments does not increase with increase in temperature above this point. Each washing was made with 500 cc. of a solution prepared by dissolving 2.5 grams of the neutral soap flake in distilled water, adding sufficient standard builder solution to give the desired concentration of the addition agent, and diluting the solution to 1 liter. The speed of the washer was kept constant at 80 r. p. m. The standard time of washing was 7½ minutes.

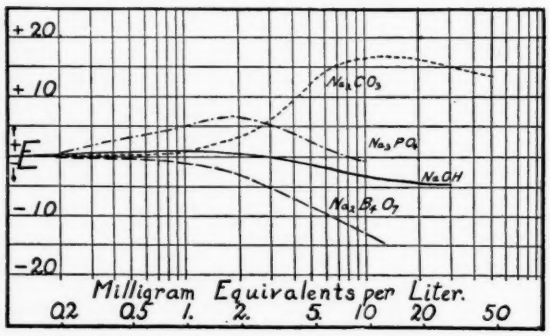

Fig. 2

Effect of builders on detergent action of soap solution.

From the results obtained in the washing tests, the "detergent effect" of each builder was calculated, by the following equation:

$$E = 100 \times (B - S)/S$$

in which E = detergent effect of builder.

B = increase in brightness of cloth (i. e., difference between the final brightness and initial brightness of soiled cloth) after five washes with 0.25 per cent soap solution to which designated amount of builder had been added.

S = increase in brightness of cloth after five washes with 0.25 per cent soap solution to which no builder had been added.

The results are shown in Figure 2 in which the values of the detergent effect are plotted as ordinates against the logarithms of the concentrations of the builders.

Discussion of Results

The addition of small amounts of sodium hydroxide to the soap solution increased the detergent action slightly. With larger quantities of the hydroxide the cleansing action was less than that of the soap solution alone. With trisodium phosphate somewhat similar effects were observed, although the increase in detergent power at the optimum concentration of the builder was much more pronounced than with the hydroxide. Analogous results were obtained with sodium carbonate, although here the increase in the cleansing action was even greater than with the phosphate. Small amounts of borate had little effect upon the detergent action of the soap; larger quantities decreased it.

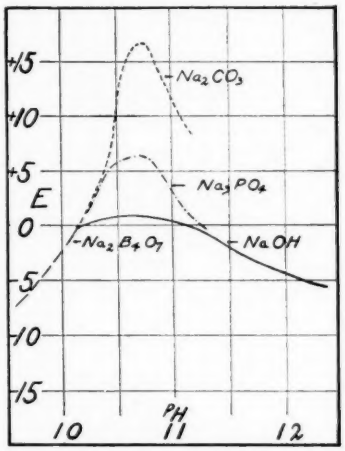

Fig. 3

Relation between pH and detergent action of various builders in soap solutions.

In Figure 3 the detergent effects of the builders, in soap solutions of the standard concentration, are shown as ordinates against the corresponding pH values of the solutions. With sodium hydroxide, trisodium phosphate, and sodium carbonate the detergent effect increased to a maximum and then again decreased. All three of these compounds showed their maximum detergent effect in a solution which had a pH of 10.7. The detergent powers of the builders at this optimum point were:

	%
Sodium hydroxide	0.6
Trisodium phosphate	6.4
Sodium carbonate	16.2

Sodium borate showed negative detergent effects at all the concentrations studied.

It is evident that the efficiency of washing does depend to some extent upon the concentration of hydrogen ions in the soap solution. Maximum efficiency is attained only when the solution has a pH of 10.7. In this respect these results are at variance with the conclusions of those investigators who have used the surface tension as an index of detergent power. There seems to be ample evidence to show that the surface tension of soap solution is decreased more or less continuously by the addition of alkalies, but to conclude from this that the detergent power increases continuously with the alkalinity is evidently incorrect.

The present results agree, qualitatively at least, with those of Vincent (12), who found that the emulsifying action of soap passed through a maximum as increasing amounts of sodium hydroxide or trisodium phosphate were added to the solution. Vincent advanced the hypothesis that the decrease in detergent power which occurs when the concentration of the added sodium compound exceeds a certain optimum value is due to the adsorption of sodium ions by the suspended particles. That this explanation is not adequate is shown by the fact that the hydroxide, the phosphate, and the carbonate all exhibit their maximum effects as builders at exactly the same alkalinity, although the concentrations of the three substances and of the sodium ions derived from them are widely different at this optimum point. If the final decrease in detergent action were due to the sodium ions,

we might expect the maximum for each compound to occur at approximately the same molar concentration and not at the same pH.

It appears that the magnitude of the increase in detergent action effected by the builder at the optimum alkalinity depends very greatly upon some specific property of the anion of the added substance, possibly upon its valence. Sodium hydroxide does not improve the efficiency of washing to the same extent as sodium carbonate or sodium phosphate. Sodium phosphate, which at the point of optimum alkalinity for detergent action is largely hydrolyzed to form univalent H_2PO_4- ions and divalent HPO_4-- ions, is much more effective; while sodium carbonate, with its divalent CO_3-- ions, shows an even greater action. It is interesting to note that when the detergent effect of borax, which in solution is hydrolyzed to form univalent and divalent ions of the type of H_2BO_3- and HBO_3--, is plotted against the pH values the curve is an almost smooth continuation of that obtained with trisodium phosphate.

Several of the investigators that have adopted the surface tension as a criterion of detergent power have observed that the sodium hydroxide has a much greater effect upon the surface tension than the carbonate or the phosphate. If the reduction of surface tension is a true criterion of the efficiency of the builder, the logical conclusion is that sodium hydroxide is more effective as a builder than any of the other alkaline compounds and should be used to the exclusion of the phosphate and the carbonate. The fact remains, however, that in most localities caustic soda is not used to any great extent in commercial laundry work. The discrepancy between theory and practice has been explained by saying that the hydroxide is too strongly alkaline—that it weakens and discolors the cloth. This does not explain why a relatively large amount of phosphate or carbonate cannot be replaced satisfactorily by a smaller amount of caustic in order to give a solution of the same alkalinity and surface tension at a lower cost. The hydroxyl ions from sodium hydroxide are not different from the hydroxyl ions formed by the hydrolysis of sodium carbonate and at the same concentration should not attack the cloth to any greater extent. The writers' results indicate that the reason for the limited use of caustic soda as a builder is that, even at the optimum concentration, it is comparatively inefficient in increasing the detergent action of soap.

The fact that the addition of borax alone to a neutral soap does not increase the detergent power does not indicate that borax is without value as a builder. Borax, when added to a soap which contains an excess of free alkali, would act as a buffer and tend to neutralize the objectionable effects of the alkali in weakening and discoloring the cloth and in decreasing the efficiency of washing. Furthermore, it is quite probable that the addition of borax to a soap solution which had been brought to the optimum alkalinity by the introduction of free caustic or an alkaline salt would very markedly increase the efficiency of the soap. The influence of various anions upon the detergent power of soap at various alkalinities is being made the subject of a separate investigation.

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THE STRIPPING OF DYED CELLULOSE ACETATE

OWING to the fact that cellulose acetate is very commonly dyed with derivatives of anthraquinone, the problem of stripping becomes a matter of considerable difficulty. For hydrosulphite and the other commonly used decolorizing agents are inefficient, and often without any action at all. It is possible to lighten a dyeing on cellulose acetate by treatment with soap at about 80° C., but a complete removal of the color is almost impossible. On the small scale good results can be obtained by the addition to the stripping bath of a swelling agent, one of an acid nature being the most efficient. Thus one can use a bath containing 5 cc. of sodium hypochlorite and 1 cc. of formic acid per liter, but this mixture cannot be used on the large scale owing to the quantity of chlorine which is evolved. Furthermore, the material is liable to acquire a yellow stain, especially in the case of mixture fabrics containing real silk.

The Rhodiaseta Company have, however, recently patented a method which gives an almost perfect stripping, without the objectionable accompaniments of the chlorine process. They propose to treat the dyed material in a bath containing 2 grms. of decolorizing charcoal and 5 grms. of soap per liter, at a temperature of 80° to 85° C., until sufficient color has been removed. The ordinary dyeing machine is quite suitable. In the case of very dark shades the operation may take several hours, but since the ingredients of the bath have no deleterious action on any textile fiber, this is not a matter of great importance. In order to obviate difficulties in the subsequent washing-out of the finely divided charcoal, protective colloids such as glucose, dextrine, or glue, may be added to the stripping bath, but excess should be avoided, as this may interfere with the action of the charcoal. An addition of from 10 to 20 grms. of gelatine per 100 liters is quite sufficient.

Textile Manufacturer.

Manufacture of Cellulose Acetate. (Relates to the art of making cellulose esters of organic acids by treating cellulose or cellulose derivatives with active acylating agents such as the anhydrids or halids of the organic acids.) Lloyd M. Burghart, of Baltimore, Md., assignor to U. S. Industrial Alcohol Co., of N. Y., N. Y., No. 1,816,564, July 28, 1931.

Practical Color-Measurement*

By **WALTER GINZEL**

THE actual measurement of color, as a development of recent date, like all other attempts to displace purely "practical" (really only empirical) working methods, and to substitute for them exact methods and devices, has gained up to the present remarkably little notice or comprehension. This is perhaps due to the fact that the business value of the matter has not been recognized so clearly as to exert an essential influence upon the customary course of the processes being carried on. Color-measurement in the exact sense has not been so clearly understood that it could force its own way on its own merits.

The requirements incumbent upon the textile industry today include a whole series of testing operations involving mechanical measurements, of a sort which may be said to belong to the "iron age" of a scientifically conducted industry. Color-measurement justifies itself in the sight of the organization of a dyeing plant, insofar as it furnishes definite figures which can provide, with other determined numerical values, the basis for any further definite evaluation of the result of the process being carried on. Thus, it is the task of color-measurement to substitute, for the usually uncertainly determined value for color-impression, a definite numerical value for the actual color, determined by some dependable measuring-device.

Of all the means of measurement devised for the determination of this numerical value, the method of Ostwald, based upon the elements of his color-theory, has proved in practice to be the most suitable. In what follows, we shall speak essentially, therefore, of the measurement of color-impression in the Ostwaldian sense, which recognizes a color quantitatively by these magnitudes: the full color V, the white-content W, and the black-content S, and qualitatively by the color-tone. The fundamental equation of his theory, valid for non-lustrous surfaces, $1_{oo} = V \times W + S$, always employs, for the construction of its color-figure, the well-known double color-cone as a construction-formula of fundamental interest; but for the requirements of practical work the determination of the color-tone, reckoned on the color-circle divided to 24ths, as well as the measurement of the black and the white content, is sufficient; and practical workers value Ostwald's double color-cone as a system of the first class in importance only on the ground that no better or more useful method has up to the present been offered.

It may be stated here that the equation just mentioned has given rise in some quarters to a complete misunderstanding of its meaning, both in practical color-work, and in other situations far removed from the field of practical dyeing; and has provoked much mental disquiet in circles which conceived that there had been placed in their hands a means of becoming completely independent of color-experts. Their bewilderment on one side went so far, that some workers undertook earnestly the task of producing a "neutral" black, which one would need only to add to a pure yellow, red, blue, or green dyestuff in the proper quantity, to form any desired mode-tone, in a simple, certain, and rapid manner. This "neutral" black, however, proved to be a myth, because the above equation depends first of all upon the laws of mixture and reflection of light, and so involves an additive procedure, while the mixing of the yellow or the red dyestuff with the dull neutral black forms a so-called subtractive mixture, and further would possess the disadvantage, that the one or the other fraction of the mixture, say, the red component, by use of different quantities at different times, might possess a stronger or weaker affinity for the fiber under the different conditions, and the purpose in view could no longer be classified as involving a "neutral" element. I have mentioned this difficulty at the first merely to dispel any possible similar misunderstanding as to the purpose of practical color-measurement, and so to disbar any ill-advised attempts to use any color-values found, as a means of producing in a predictive way, so to speak, new mixtures of desired qualities.

In the textile industry, and especially in the finishing industry (taking this term in its broad sense, and so including the processes of dyeing), practical color-measurement has made its way only in the following field:

1—Testing of the degree of bleaching in white-bleaching.

2—Testing of dyestuffs for purposes of systematic selection.

3—Testing of the light-fastness of dyestuffs.

4—Colorimetric investigations.

5—Measurement of luster of white or colored surfaces.

6—Production of general color-cards for statistical or propaganda purposes, by use of the Ostwald color-tone triangle.

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* Translated from Seide 36,161 (1931).

7—Production of a rational dyeing procedure for the various materials within the scope of the color-triangle dyeing possibilities (establishment of a "region of tolerance" for fast dyeings).

On account of its adaptability for rapid service and simple manipulation, the Pulfrich Step-Photometer, manufactured by C. Zeiss, and familiarly referred to as the "Stupho," has won a high place for itself in the practical carrying out of color-measurements; we do not mean to hint that there are no other good instruments available. Reference needs to be made only to the polarization-color-mixture apparatus of Ostwald, called the "Pomi," and to the half-shadow photometer of Janke and Kunkel, of Cologne.

The estimation of the numerical value of the observations of a colored surface can first of all be carried out with the idea of locating the plotting-points of the color-tone measured upon the Ostwald triangle or double cone, the axis of which forms a rectilinear gray-scale, divided into even steps or graduations, with W (white) at the upper, and S (black) at the lower, extremity. The common base of the double cone forms a color-circle, which along its circumference bears divisions allotted to the various colors in sequence, from yellow through orange to red, purple, blue, ice-blue, sea-green (blue-green), leaf-green (yellow-green), and again back to yellow.

For this series of the bright colors—bright in contrast to the dull ones of the gray series—Ostwald chooses the simplest conceivable form, a circular line, and chooses this of the simplest conceivable order, since the gray line stands vertically upon the center of the circle, on the circle's plane, by which it, as the axis of the double cone, is bisected. Upon the surface of the upper cone, whose apex is W, and whose base is the color-circle, lie the "light-clear" colors, and upon the surface of the lower cone, with the apex S and the same base, the "dark-clear" colors, the first theoretically designated as "black-free," the second as "white-free." The "dull" colors are located within the cone.

The color-circle itself Ostwald divides into 24 parts, according to determined differences or steps in difference of tone, of the same magnitude, beginning with yellow as 1; and lays off through each of these a plane passing through the line (axis) WS, and upon the so formed "color-tone-similar" triangle VWS the transitions of the color V into the gray tones. Upon the parallels to VW, drawn through the separate steps or divisions, lie the "black-similar" colors; upon the parellels V to VS the "white-similar," dull colors, and upon the parellels to WS the "pure-similar" colors.

The procedure according to which Ostwald determines the black-and the white-content is founded upon an observation made by Lambert, that a surface appearing pure white in daylight appears under monochromatic light as a monochromatic surface, of the same color-tone as the source of illumination. If, for example, we observe a red rose beside a barium-white plate (the purest white known), through a red-glass filter, the rose, in comparison with the barium-white plate, will display a plainly recognizable blackish, dull tone. The reason for this is, that the color-effect of the red rose, obscured by daylight, is produced by absorption of daylight, which nullifies at least the green and part of the yellows, and also depresses more or less the intensity of the red, violet, and blue radiation. That part which in this manner is lost, that brilliancy in the spectral area characteristic of the colored body when viewed under ordinary conditions, is what, according to Ostwald, we call the black-content of the color. This black-content is measured by Ostwald through an adjusted filter, which transmits the spectral region normally emanating from a colored body.

For the measurement of the white-content is employed a colored filter which transmits a spectral region different from that of the colored body, and completely absorbs or extinguishes the color of the body, or else transmits only those rays which do not reflect the red of a colored body. Observed through this counter-color filter, a green one, an ideally red body-color must appear absolutely black.

Since color-measurement consists in nothing other than the determination of the degree of reflection of light, designated as remission, then the remission curve obtained by spectroscopic measurement, or by use of the 7K filter of the step-photometer, will give visible information of the ratio between the white-and black-content of a colored surface. In this curve, the white content is expressed as a minimum, while the brightness measured with the adjusted filter is the maximum.

By the term full color, Ostwald designates that color (according to his phrase "color-half," or better, "spectrum-half," which comes into visibility through the combined action of the whole spectral area extending over one-half of the whole spectrum; it is not synonymous with the phrase color-tone, although in practice one very often meets with this confusion of the two in citations of the formula $1_{oo} = V + W + S$.

The color-tone is determined only through its quality, since its fractional value in the total color-reflection is involved in the above formula.

The brightness, measured with the help of the supplementary filter, also gives directly the white-content W, according to Ostwald; the brightness, if measured with the adjusted filter, gives the black-content S through the formula $S = 1_{oo} - h$.

It is now necessary to mention an important psychophysical law discovered by Fechner, namely, that between stimulus and response such a relation exists, that the stimulus must be graded down in geometric or logarithmic progression, in order to cause the response to diminish in arithmetical progression. The gray series of the Ostwaldian equal-color-tone triangle, divided into equal parts, is in accordance with this law.

It therefore became the task of the optical physicist to provide a photometer which should be, as accurately as possible, adapted to the distinctions in sensitivity to-

ward color-difference, which would be simpler, easier to manage, and not so expensive as the instruments constructed for purely scientific investigations, and which, however, would have the advantage that, independently of any arbitrarily-chosen and frequently impermanent standards, it would make possible the direct measurement of the relations between the optical properties of materials under comparison. Such a photometer was constructed by Prof. Pulfrich at the Zeiss works at Jena, and, as already stated, has been given the name of the Step-Photometer, or "Stupho."

One of the first fields of practical work into which this sort of measurement technique has made its way successfully, is that of cotton white-bleaching, which involves working with so-called degrees of bleaching, as ¼, ½, ⅝, ¾, and 4/4, that is, with wholly meaningless concepts of value, since they are of arbitrary origin. Through the white-content numbers obtained with the Step-Photometer (for these numbers are essentially what the older concepts come down to), we obtain a more exact measurement of the degree of whiteness gained by the bleaching process. Besides, this measurement gives a convenient means of estimating the progress of the bleaching, and affords valuable help for a really accurate bleaching operation, in that the bleaching is known to be ended as soon as the white-content of a sample of the goods corresponds with the standard white adopted as the standard. If, during the progress of the bleaching, one notes graphically, at exact intervals of time, the increase in the white-content, then the duration of the bleaching-process can be read off, on the graph, for a desired degree of bleaching of other lots, if the same conditions are maintained.

An essential enrichment of the range of ordinary methods of testing has been brought about in the subject of the systematic choice of dyestuffs, so important for the rational conduct of the dyeing industry; color-measurement is able, through this means of really exact measurement, to establish the relation between the power of exhaustion or bath, and cost, of competing dyestuffs which are similar in tone, with reference also to their fastness properties.

The method, formerly and still now prevailing, consists of so-called comparative test-dyeings, a standard product, kept in stock, and the competing dyestuff under examination, are each dyed at equal strength, usually 2%, upon the same material, with maintenance of exactly the same conditions—additions, bath-volume, temperature, etc.,—if the competing dyestuff gives a stronger, fuller dyeing, the dyeing with it is repeated with gradually and regularly diminishing percentages of dyestuff, 1.8, 1.7, 1.6%, etc., and the results compared with the standard 2% dyeing, until a percentage is reached which gives an exact match. If the dyeing is weaker, then the percentage is gradually raised above 2% in regular sequence, until a match is obtained. A simple calculation then gives the relative value of the competing as against the standard dyestuff. This method, however, does not give the dyeing power of the dyestuff in any complete sense, and has also the disadvantage that, for the attaining of the point of equal dyeings, many repeated dyeings with the competing dyestuff have to be carried out.

The method of comparative dyeings carried out exclusively with definitely measurable values makes it possible, in the shortest and most exact way, to assign the relation of dyeing power of two dyestuffs similar in tone, and through graphic expression of the comparison of values, to set down as a permanent record a single test of permanent value.

The method now being cited requires only a parallel series of dyeings, of the standard and of the competing dyestuff, made in steps; that is, maintaining the same conditions, dyeing a series of swaches with amounts of dyestuff decreasing geometrically through 8 stages. In each one of these dyeings the white content is determined as the measure of exhausting power, for the fuller the tone, the lower the white-content. The values so measured are plotted upon cross-section paper in such a way that the white-content figures are laid down upon the logarithmically divided ordinates, and the dilution-values, in percents, upon the similarly divided abscissae. For each dyestuff is thus obtained a white-content curve, which will permit a comparison of values by the following procedure: from several of the points corresponding to the dilution-series of the white-content curve of dyestuff A are drawn parellels to the abscissa, and intersecting the white-content curve of dyestuff B. The intersections, produced, give, through projection upon the logarithmic scale of the abscissa, those quantities of dyestuff B, which dye the same white-content as the selected dilution-stages of dyestuff A. Thus, for any desired quantity of dyestuff A, the corresponding quantity of dyestuff B for the same degree of exhaustion is obtained.

It is then merely necessary to know the cost of each dyestuff, in order to gain quickly and completely the desired information regarding the dyeing-power and cost-value of the dyestuff under consideration.

Where it has been employed, this method of measurement of the comparative value of dyestuffs has been brilliantly successful in modern practice, and the results obtained have been recognized as useful and exact in the highest degree, on the ground of exactness and of correspondence with the more clumsy and older method of trial dyeings. The method absolutely and conveniently fulfills the requirements which are made upon a scientific management of the industry; and an industry which builds up its stock of standards by the above method may properly advance the claim that it is being conducted along rational and systematic lines.

As the next practical field of color-measurement mastered by the method which we are considering, we may cite the testing of light-fastness of dyestuffs in the dyed state, if we can judge of its frequent mention in the journals. For the determination of the fastness to light, there has been up to the present no absolute method

available; and the degree of 1—8 (8 as the highest value), which has been established by the German Fastness Commission, permit only approximations to these degrees. We cannot now enter upon the details of the application of the color-measurement method as employed in this field, but give only the principles involved.

For uniform comparison, the testing of light-fastness of dyeings is undertaken upon such dyeings as have been prepared with a white-content of 10% (the proposal of Ziersch) upon suitable material. This value corresponds to a color-impression ranging from bright to medium. These dyeings are now exposed to the influence of daylight or sunlight, and the light-energy absorbed is measured by means of the "exposure paper" first introduced by Prof. Krais (paper coated with a light-sensitive dyestuff, Victoria Blue R, precipitated upon kaolin). Thick absorbent paper stained with Brilliant Green can also be used, with equally good results. By means of a scale prepared by exposure of such a paper under strong June sunlight for various periods, the number of "fading hours" during which the dyed sample and strips of the test-paper have been exposed side by side can be determined.

In the dyeing laboratory of the Textile Finishing Association at Crefeld, for the purpose of testing light-fastness in a way which would be independent of the greatly variable intensity of daylight, a 2000 watt lamp has been employed, so arranged that the customary sheet metal sheaths containing the swatches are set in a circle about the lamp at a distance of 15 cm. from the filament, the whole arrangement being contained in a sheet-metal housing provided with a ventilator, which draws through sufficient cool air to keep the samples from being scorched by the heat of the lamp (the whole arrangement, we suppose, is much like that of our familiar "Fadeometer"). The intensity of radiation of this lamp, tested by comparative exposures, gives in 1 hour the effect of 2 hours of exposure in bright noon sun in June.

This constant light-source makes possible an objective determination of light-fastness, if made in conjunction with the system of measurement under discussion. The determination can be made by use of the following principles. First of all, the quantity of dyestuff present, read off from the white-content curve of the dyestuff (a quantity which dyes to a white-content of 10% on a corresponding material), makes possible a rapid carrying out of comparative dyeings. These are now exposed for a given number of "sun hours," for example, 100; and we measure the increase in white-content after every 5, 10, 20, etc., hours, for this value alone is what affords the most visible criterion of alteration of tone. The results can now be plotted graphically, by introducing them into a coordinate system, in which the white-content values are located upon the logarithmically arranged ordinates, and the corresponding exposures upon the logarithmic abscissae. Or, we can evaluate the figures for white-and black-content upon the psychologcial triangle of Ostwald as follows: the location of the color on the triangle is first determined, and this is combined with the gray-plane, which with a white-content of 85% will on the average correspond to the color of the undyed material. The so obtained magnitude would theoretically be required to designate the color under a continuous exposure to light. According to an exposure of 100 hours, assumed for the determination, the values obtained would locate a definite point in the triangle, and by connecting this with the point of origin of the unexposed dyeing, a new line would be produced. This line is now drawn against the first mentioned magnitude (graphically, a line), (it can simply be measured in millimeters), and the result is an absolute numerical value expressed in percentages; the degree of fading $A = \frac{100AB}{AR}$, a result which corresponds favorably with the result as gained by merely ocular estimation.

Another method, which was first employed by Jost and Fluetsch, determines the so-called "half-value-time of fading"; that is, the time of exposure at the close of which the half-fading-value of the distance AR on the triangle is reached. This half-value-time is accordingly a measure of the resistance to fading of the dyeing, and allows the dyer to predict to some extent the probable permanence of the tone.

The above methods for testing the relative values of dyestuffs and the exact degree of light-fastness, which have been made possible by use of the Ostwaldian color-constants and the Step-Photometer, have been selected at random from the whole field in which such methods have been found useful. They quite plainly show themselves to form a decided step forward, and a decided enrichment of the field of really exact methods of true measurement.

The Dyeing of Wool-Silk Unions

By Dr. Ing. A. Foulon

(Note—Although the subject-matter of this paper is familiar to many of our readers, yet, from several inquiries recently received regarding difficulties in the process, it seems worth while to reprint the paper in English, in the hope that it may be of service to some.)

WOOL-SILK unions are, of course, weaves which combine the two fibers. A weave of cotton and silk is commonly designated as half-silk, but wool-silk is also sometimes so called. The best known wool-silk weaves are the goods known as "Gloria"; others are half-silk cashmere, bombazine and poplin.

If boiled-off silk has been used in the weaving of the wool-silk mixture, one can go ahead with the dyeing according to the methods given below. But, if the weave contains raw silk, the goods must be boiled off before

dyeing. According to the method of Moyret, this is carried out by passing the goods through a very dilute, lukewarm, hydrochloric acid bath, rinsing, and debasting the silk in a strong soap-bath at about 90° C., then rinsing in a weak soda bath, and then in pure water.

In reference to the behavior of wool-silk goods toward dyestuffs, it is to be noted that in most cases both kinds of fiber are to be dyed, though not infrequently in somewhat different shades. Any dyestuff which dyes silk will, of course, dye wool also. But not every dyestuff which dyes wool will also be taken up by the silk, so that in a given case it may happen that only the wool is dyed, and the silk remains white. Wool-silk mixtures can therefore be dyed in a distinct two-color effect, if desired. The silk is then usually dyed cold, the wool usually at the boil, so that, if we dye at the ordinary temperature, the wool remains almost uncolored when the silk is fully dyed. Thus, by due regard to the temperature of the bath, we can produce at will a difference in the shades dyed. According to the shade wished for each fiber, and the classification of effect, whether, e. g., for "uni" (solid) o r "changeable" (two-color effect), we shall have to choose for the dyeing either such dyestuffs as color wool and silk each, evenly, under certain conditions, or those which do not dye, or hardly dye, the silk.

The wool can be dyed with acid, substantive, or basic colors. Mordant dyestuffs are not used with this material. For solid dyeings on wool-silk, it is best to employ such acid dyestuffs as possess a fairly similar affinity for the two kinds of fiber, so as to be able to dye evenly each of them, simultaneously, at the boil. The acid dyestuffs are the ones usually considered to be most suitable, but the substantive dyestuffs also are often suitable, especially for solid dyeings. The basic dyestuffs are used mostly only for two-color effects.

Methods of dyeing are various. Solid tones are dyed, according to one procedure, in a bath prepared only with sulphuric or acetic acid and the necessary dyestuff, while another procedure requires a bath made up with sulphuric acid and Glauber salt, or tartar preparation and crystallized Glauber salt, or acetic or formic acids and Glauber salt, or even with Glauber salt alone (with, of course, the dyestuff).

In what follows, we describe briefly a few methods, and, first of all, for solid or uni-colors. The wetted-out goods are entered into a bath prepared with the necessary amount of sulphuric acid and dyestuff, the temperature raised slowly to boiling, and the bath kept at the boil until the wool and silk show approximately the desired depth and shade of tone. If the silk is not full-toned enough in comparison with the wool (and this may be the case, especially with darker dyeings), it is dyed to match by lowering the temperature and adding more dyestuff. If it is necessary to shade the silk with a basic dyestuff, in order to gain brightness and fullness of tone, the first bath is run off and the silk redyed in a new bath with more acetic acid, at the necessary temperature. Finally the goods are rinsed. The "Agfa" (I.G.) procedure is similar; the first dyeing is carried out at the boil in a bath containing acetic or sulphuric acid, and the rest as above. Both solid and two-tone effects can be gotten in this way.

For two-tone effects, the following methods are best suited. The wool is first dyed at the boil in a bath weakly acidified with acetic acid. Then to the bath are added amounts of acetic or sulphuric acid, at first very little, but gradually increasing, until the bath is almost exhausted, which will generally be the case after two hours. Instead of acetic acid, 5-10% of tartar preparation may be used. Then the silk is overdyed directly, or, according to the kind of shade desired, dyed after previous stripping, with ammonium acetate, or one of the assistants noted below, in a fresh, warm bath acidified with acetic acid, at 30° C., with addition of basic or acid dyestuffs, as desired.

Although many dyestuffs, used according to the above method, leave the silk fairly light in tone, or even clean, yet in some cases the whiteness of the silk is not quite satisfactory as such, or for the production of certain shades. In that case, after the first dyeing, we boil the goods for 10 minutes in a stripping-bath containing ammonium acetate, in order to get a pure white, the operation in some instances needing a repetition. In place of ammonium acetate, we can use ammonium oxalate (Cassella and Ciba) for the stripping. If very soft water, or condensation-water, be used, the addition of ammonium oxalate may perhaps be omitted. If, when stripping with ammonium acetate, which acts more powerfully than the oxalate, the wool also loses too much of its color, then the stripping has been too lengthy, or the concentration of the bath too high. Further stripping procedures after the first dyeing involve the treatment of the goods with a Turkey-red oil or hydrosulphite bath.

Instead of working with a multiple-bath procedure, we can also employ the single-bath method (Badische). In this case, there is added to the bath 10-20% of acetic acid and the necessary amount of acid or basic dyestuff, the goods are entered at 30° C., the bath raised to the boil, the boiling continued until the wool is dyed to the desired depth, and the steam shut off, the dyeing being allowed to go on in the hot bath to the desired final point. The shading of the silk, if such is necessary, is done with additions of basic dyestuff, below the boiling point. But we gain purer effects if the wool dyestuff is put on at the boil, the steam shut off, and the silk after-dyed at a comparatively lower temperature by addition of the basic colors.

The pre-dyeing of the wool (see above) can also be carried out by making up the bath with 10% of crystallized Glauber salt, 8-15% of 30% acetic acid, 1¼-2½% of formic acid (85%), and the necessary amount of dyestuff. The goods are entered directly into the boiling bath, and boiled at least one hour. After the rinsing, the goods are soured with an acetic or formic acid bath.

Wool-silk goods may also be dyed with chrome-developed colors, with addition of 10% of acetic acid and 10%

of Glauber salt, since these dyestuffs leave the silk practically untinged. The goods are entered at the boil, and the boiling continued for one hour. Three per cent of potassium chromate is then added, and the shade developed by further boiling. To whiten the silk, a bath of ammonium acetate is generally used, or the goods are simply passed through a hot soap-bath.

Which dyestuffs will be most suitable for a particular case, the dyer can decide from the sample cards of the various firms.

Translated from Monats. f. Textil-Ind. 45,447 (1930).

New Patents

Process for the Preparation of 2-Amino-Beta-Phenyl-Anthraquinone by Ring Closure. (Dyestuff intermediate, which comprises heating 2-amino-para-phenyl-ortho-benzoyl-benzoic acid with sulfuric acid as a dehydrating agent in the presence of nitrobenzene.) Philip H. Groggins, of Washington, D. C.; dedicated, by assignment, to the Government and the People of the U. S. A., No. 1,814,149, July 14, 1931.

Dyestuffs Containing Chromium and Process of Making. (Consists in treating an azo dyestuff free from chromium, but containing chromable groups with a chromium compound.) Karl Holzach, of Ludwigshafen-on-the Rhine, Hans Kammerer, of Mannheim, and Werner Muller, of Ludwigshafen-on-the-Rhine, Ger., assignors to General Aniline Wks., Inc., of N. Y., N. Y., No. 1,814,218, July 14, 1931.

Manufacture of Viscose. (Comprises wetting sheets of raw material with a steeping liquor and permitting the reuse of 100% of the caustic liquor formerly wasted.) Galo W. Blanco and Carleton Henningsen, of Buffalo, N. Y., assignors, by mesne assignments to duPont Rayon Co., of N. Y., N. Y., No. 1,814,542, July 14, 1931.

Manufacture of Viscose. (The method of shortening the time of ripening of a viscose solution which comprises adding hemi-celluloses thereto.) Galo W. Blanco and Carleton Henningsen, of Buffalo, N. Y., assignors by mesne assignments, to duPont Rayon Co., of N. Y., N. Y., No. 1,814,543, July 14, 1931.

New Azo Dyestuffs and Process of Preparing Them. (For example, consists in diazotizing 4-nitro-2-amino-1-methylbenzene and coupling with the sulfonic acid prepared by sulfonating 2-hydroxynaphthalene-3-carboxylic acid (5'-chloro-2'-methyl-1'-) anilide; a ruby-red dyestuff difficultly soluble in water and which can be made up in the usual manner into a color lake.) Otto Sohst, of Hochst-on-the-Main, Ger., assignor to General Aniline Wks., Inc., of N. Y., N. Y., No. 1,815,133, July 21, 1931.

Fast Sulphur Dyestuffs (Dyeing Bluish to Green Shades) and Process of Making Same. (Obtainable by causing a sulfu[illegible]g agent to act upon 2-(4'-hydroxy-aryl-amino)-6-aryl-amino-naphthalenes.) Richard Herz, of Frankfort-on-the-Main, and Wilhelm Hechtenberg, of Frankfort-on-the-Main-Fechenheim, Ger., assignors to General Aniline Wks., Inc., of N. Y., N. Y., No. 1,815,144, July 21, 1931.

New Azo Dyestuffs and Metal Compounds Thereof. (Dyeing wool yellow shades, obtained by coupling the diazo compounds of aminosulphosalicylic acids with such phenols as are substituted in the para position to the hydroxy group.) Hans Krzikalla and Hans Kaemmerer, of Mannheim, Ger., assignors to General Aniline Wks., Inc., of N. Y., N. Y., No. 1,815,782, July 21, 1931.

Praparation of Mordant Disazo-Dyestuffs (of Orange Shades). Ernst Bodmer of Basel, Switzerland, assignor to Durand & Huguenin S. A., of Basel, Switzerland, No. 1,815,827, July 21, 1931.

whereas soap is extremely sensitive and causes precipitations which are a constant source of all sorts of trouble in the textile industry; even lime soap already formed on the fiber is dissolved by Igepon A.

Complex Chromium Compounds of Azo Dyestuffs.—(Obtainable by coupling diazotized 4-nitro-2-amino-phenol-6-sulfonic acid and acetylacetone, dyeing wool yellow shades with a reddish tinge.) Hans Krzikalla, of Mannheim, and Franz Bluemmel, of Seckenheim, Ger., assignors to General Aniline Wks., Inc., of New York, N. Y., No. 1,812,345, June 30, 1931.

Manufacture of Valuable Products from Lignin Sulphonic Acid (Sulphite Cellulose Waste).—(In combination a dyestuff paste or powder and the product of the moderated oxidation of sulphite cellulose pitch with nitric acid, the said combination being characterized by ready dispersibility in water.) James Baddiley, Arnold Shepherdson and Anthony James Hailwood, of Blackley, Manchester, Eng., assignors to British Dyestuffs Corp., Ltd., of Manchester, Eng., No. 1,812,675 June 30, 1931.

Vat Dyestuffs of Anthraquinone Series

German Patent No. 448,286 (16/VIII/'27)—*I. G. Farben-Ind. A.G.*—The compounds cited are complicated alpha-amino-anthraquinones of a simple general nature. Alpha-amino-anthraquinone or its derivatives is either treated first with an aromatic acid, its anhydride, or chloride, to form an aroyl-amino group, and then with dibasic acids (if an aliphatic acid, it must contain more than one chain-carbon, if an aromatic acid, any suitable one will suffice), their chlorides or anhydrides. Or, the amino-anthraquinone is first treated with the dibasic acid or its derivative, and then with the simple acid or its derivative. Evidently the process consists in a doubling up of two anthraquinone molecules through linkage by the nucleus of the dibasic acid, the other hydrogen of each so substituted amino-group being replaced by the simpler acid radical. The character of the dyestuffs so produced is claimed to be essentially different from that of the simpler ones produced by acidylation of amino-anthraquinones by monobasic acids; the dyeing power, the strength, of the dyestuffs is greater relatively, and the depth of tone is greater. The products show excellent fastness toward boiling, kiering, and light.

Technical Notes from Foreign Sources

Determination of Unsaponified Neutral Fat in Soap

Dr. O. Schuette—Monats. f. Textil-Ind. 45,439 (1930) (*Abstr. from Seifensieder Ztg.*, 1929, *p.* 245).—A proposed modification of the standard method followed in Germany. Its details are as follows: 20 g. of the soap are very finely powdered (this is important), placed in an Erlenmeyer, and covered with a mixture of 80 cc. of 95% alcohol and 70 cc. of water, in the latter 1 g. of sodium bicarbonate being dissolved before adding the alcohol. The soap is dissolved in the dilute alkaline alcohol by immersing the flask in a warm water bath (not over 50° C.). The purpose of the bicarbonate is to neutralize any free caustic possibly present, as it comes into solution, thus preventing the least hydrolysis of the fat which is to be determined: it seems to work better than the usual addition of 1/10 N sulphuric acid for the same purpose. After solution has taken place, the liquid is cooled to about 20° C. (not lower, since solid soap may separate) and extracted 3 times with about 70 cc. (each portion) of petroleum ether of b. p. 30°-50° C. The united extracts are allowed to stand a short time, to allow any soap taken up to separate; if much soap does separate, the united extracts are to be filtered into another separatory funnel. To remove traces of soap, and of free fatty acids possibly taken up by the petroleum ether, the extracts are shaken out with a mixture of 15 cc. of 1/10 N soda solution (Grosser, Seifensieder Ztg., 1929, p. 128) and 15 cc. of alcohol, and then 3 times with about 30 cc. (each portion) of 50% alcohol. The petroleum ether extracts are now filtered through calcined Glauber salt into a weighed Erlenmeyer flask of about 300 cc. capacity. The separatory funnel and filter are to be rinsed several times with petroleum ether into the Erlenmeyer. The Erlenmeyer is attached to a condenser, and the petroleum ether carefully distilled off, and the flask, after being blown out, set in the steam drying-closet to stand at 100° C. for about 1¼ hours. The weight gives the total of free fatty acid, unsaponified fat, and the unsaponifiable matter, by multiplication of the weight by 5, the figures express the percentage. This weighed residue is dissolved in 20 cc. of neutralized alcohol, and titrated for the free fatty acids with 1/10 N alcoholic caustic. The figure obtained is reckoned as oleic acid, in percentages of the soap. Then ½ cc. of an aqueous 10 N caustic potash solution is added, and the neutral fat saponified (water bath and reflux condenser) by ½ hour's heating. After 18 cc. of water have been added, the mixture is cooled, and the unsaponifiable matter extracted with 3 30 cc. portions of petroleum ether. The united extracts are washed by shaking out 3 times with 50% alcohol. The petroleum ether, which now contains only the unsaponifiable matter, is filtered as before through calcined Glauber salt, with rinsing as before, and the solvent distilled off. The weight of the residue gives the unsaponifiable matter, which, as before, needs only multiplication by 5 to be expressed as percentages. The percentage of unsaponified, neutral fat is easily calculated from the figures obtained.

Determination of the Ferricyanide Number

Prof. Freiberger—Monats. f. Textil-Ind. 45,440 (1930).—Another analytical paper, of use particularly to those working with bleaching or mercerizing.

The standard solutions are as follows:

a—Ferricyanide solution, 1/200 N. . 1.65 g. of c.p. ferricyanide are dissolved in 1 liter of distilled water, the solution being kept in an opaque bottle, or in one covered completely (even the bottom and stopper) with black paper, so that light is absolutely excluded. It should be tested from time to time by titration with the "thio" solution, which itself is checked against KIO_3.

b—Iodate solution, 1/200 N. . 0.1783 g. of pure potassium iodate are dissolved to 1 liter in distilled water. This serves for standardizing the other solutions.

c—Starch solution. 1 g. of soluble starch is dissolved in 100 cc. of an aqueous, saturated solution of pure NaCl. This solution does not keep well, of course.

d—Zinc solution. 50 g. of crystallized, pure zinc sulphate are dissolved, and made up with 250 g. of pure NaCl to 1 liter.

e—Thiosulphate solution (approximately 1/200 N.) . 1.45 g. of pure sodium thiosulphate are dissolved in distilled water, and the solution allowed to stand in a flask of dark glass for about 14 days. It is then titrated several times against the iodate solution, and its normality factor designated by f. The titration is carried out by acidifying exactly 20 cc. of the iodate solution with 10 cc. of 10% sulphuric acid, and treated with a few small bits of potassium iodide; after these have dissolved, a few drops of starch solution are added, until the mixture is blue, and the whole then diluted to 100 cc. It is titrated to decolorization with the thio solution, the vessel being placed upon a white substratum. The normality factor f is to be reckoned as a dividend from 20 and the quantity of this solution in ccs., therefore 20, through the quantity of this solution in ccs., found by titration.

Procedure (direct): suitable for cotton, linen, other little lignified, not strongly broken down, or impure fibers, and for bleached cell-substance.

The material is disintegrated; fabrics are cut into squares of about 9 mm. across. Of the material, 0.2 g. or less (in case of a high content in reducing substance, only 0.1 g.) are placed in a wide testtube of resistant glass, diameter 30 mm., height 150 mm., and 3-4 clean

glass beads are added (carefully cleaned, by washing with ether-alcohol, dilute sulphuric acid, then with a warm ferricyanide solution, finally with distilled water, with which also the beads must be washed between each of the preceding cleasing agents). 10 cc. of ferricyanide solution and 10 cc. of 10% c.p. caustic soda solution are then added, the testtube placed in a waterbath, in which the water is boiling, and kept there for exactly 10 minutes, shaking gently about every 2 minutes. It is then removed, cooled down rapidly, and the contents treated with 2 cc. of 30% c.p. acetic acid. A few grains of potassium iodide are then added, and, after they are dissolved, 10 cc. of the zinc solution, exactly 5 drops of the starch solution, and the mixture then titrated to decolorization with the thio solution. The amount of the thio solution used is designated by t. Check determinations of t should not vary by more than 1%. The reduction-number expressed in terms of thio solution is T-t. On account of the variability of normality of the thiosulphate solution, its factor f must be determined (see above). (The factor f = 20 divided by the number of ccs. of the thio solutions used.)

In reckoning in this factor f, the formula (T-t)f gives the desired amount of 1/200 N ferricyanide solution which expresses the reduction-number, but only after its calculation upon 100 g. of cellulose. Expressed in solid ferricyanide, the ferricyanide number = f, the amount of ferricyanide needed for 100 g. of substance. The procedure gives exactly concordant results. Only 20-25 minutes are necessary for carrying out the analysis, if the standard solutions are at hand.

Procedure (indirect): by boiling-off the material. This method is best suited for substances of high content in substances reducible in alkaline solution. The results check well, but the ferricyanide numbers are somewhat higher than those obtained by the direct method of determination. One g. of the substance under examination, or, in case of a substance with high content of impurity, less than 1 g., disintegrated as before, is heated for ½ hour with 150 cc. of NaOH of 5 B°., in an Erlenmeyer flask, closed by a glass ball resting lightly upon the mouth of the flask. The flask is heated in a boiling waterbath; during the heating, the flask is now and then gently shaken about, the cellulose not being thereby shaken above the surface of the caustic solution. The flask and contents are then cooled in water, the contents filtered through a Jena glass-filter of sintered glass No. 1, and the filtrate brought to 250 cc. with distilled water. The residue is boiled out again, in the same way as before, and treated as before, so that several extracts are obtained. The process is continued until, on titration of each extract, no difference in the amount of ferricyanide solution used is detected. Each of the extracts, therefore, is titrated, and the sum of the results gives the ferricyanide number. For technical purposes, the first titration is generally sufficient; if exact results are required, the second and third titrations, with the first, are usually enough. The titrations are carried out as follows: 10 cc. of the boil-off liquor are mixed with 9 cc. of ferricyanide solution in a wide testtube as already described, and this immersed in a boiling waterbath for 10 minutes. All else is as before.

The oxygen number: this expresses how much oxygen would be used up by 100,000 g. of substance in the titration. It is calculated from the ferricyanide number, the equivalent of the ferricyanide (658), and that of oxygen (16). Thus, the ferricyanide number 3.30 corresponds to an oxygen number of $3.3 \times 16 : 658 \times 1000$, or, more simply, $3.3 \times 24.3 = 80.2$.

For the titrations are used long burettes of only 10 cc. capacity, therefore narrow in cross-section, and carrying graduations to 1/50 cc. Distilled water is always used.

The rapid, simple, and certain nature of this analysis has aroused great interest in practical circles. A few examples are quoted, in which the ferricyanide numbers indicate the content in compounds or impurities which exert a reducing effect in alkaline solution: raw cotton weave, 11.830; viscose yarn, 13.002; acetate silk, very high; cotton weave kier-boiled, 1.410; same material, soured, 2.244; same material, chlored, 2.610; same material completely bleached, 1.625; linen yarn boiled out, 28.34; pectin from cotton, 48; soluble starch, 177; albumen, 536.

The figures obtained by this method of working agree better with the copper numbers from the same material, than with those obtained by the permanganate method. Through its speed and its exactness in results it seems well worth adoption in both technical and purely research work.

Dyeings Fast to Rubbing (Animal Fibers)

German Patent No. 453,939 (*23/III/'26*)—*Durand Huguenin A.-G.*—An earlier patent, No. 447,731 (*21/-VII/'25*) covered the use of fuller's earth for improving the fastness as above, by addition of the earth to the acid dyebath (dyestuffs used were ester salts of leuco vat dyestuffs). This improvement employs the fuller's earth in the oxidizing bath instead of the acid dyebath, and the results are claimed to be much more satisfactory. An example is given, of the dyeing of Indigosol O. There seems to be no particular departure from the ordinary mode of dyeing, save in the presence in the oxidizing bath of 25% of fuller's earth on the weight of the wool.

Printing of Azo Colors

German Patent No. 451,049 (*23/XII/'24*), *released 29/IX/'27*)—*I. G. Farben-Ind. A.-G.*—A development of G.P. No. 446,541. A thickened mixture of a diazotizable amine, a suitable second component, and nitrite, is printed upon the fabric and dried. On subsequent treatment with a bath of acid or of an acid salt, the formation of the dyestuff upon the fiber is brought about. No separate diazotization is needed, no stable diazo-mixture has to be used, and steaming and hanging are unnecessary. The process works well for mixed ones also. Further, the printed goods can be stored before development. Four examples are given.

Leave of Absence

Mr. H. H. Willis, Director of the Clemson College (S. C.) Textile Department, announces that Dr. C. E. Mullin, Professor of Textile Chemistry and Dyeing and Head of the Textile Chemistry and Dyeing Division of the Textile Department of Clemson College, has been granted leave during the session 1931-1932. Dr. Mullin plans to travel in South America and in Europe during the coming year.

Mr. H. H. Willis, Director of the Clemson College (S. C.) Textile Department, announces that Dr. H. L. Hunter has been elected to act during the coming year as Head of the Textile Chemistry and Dyeing Division of the Clemson Textile Department. Dr. Hunter received his training in chemistry at Cornell University, receiving his Doctor's degree from that institution in 1928. Part of his work at Cornell was done under a Heckscher Research fellowship. He is a member of Sigma Xi, national honorary research society, and Phi Kappa Phi, national honorary scholastic fraternity.

Dr. Hunter has had several years' teaching experience and for the past two years has done considerable work in the Textile Chemistry and Dyeing Division of Clemson Textile Department toward a Doctor of Science degree. Under a special cooperative agreement, credits for certain graduate work in the Clemson Textile Department are accepted by the University of Nancy, France, toward a Master's or Doctor's' degree at that institution.

New Course

Realizing the greater emphasis that needs to be placed on textile distribution compared with manufacture against former years, the Lowell Textile Institute has begun the development of courses of study in textile merchandising. There is now being offered for the first time a complete Sales Option course which has been planned so that it will correspond to the general textile option or one of the manufacturing options in the Textile Engineering degree course. The degree of Bachelor of Textile Engineering is awarded for the completion of this four year course. Last year the Institute added to its staff Mr. C. F. Edlund, a graduate of M. I. T., who has started regular instruction work in Marketing courses. Mr. Edlund is spending this summer in New York City doing further study at Columbia, and also spending considerable amount of time in the textile market, meeting people and discussing methods now in use.

Research Laboratory

H. H. Willis, Director of the Textile Department, Clemson Agricultural College, Clemson College, S. C., announces that the new and up-to-date textile testing laboratory of the Department has just been selected by the U. S. Institute for Textile Research, Incorporated, as a "participating laboratory." Only three other laboratories in the United States have thus far qualified for this particular type of research work.

Sulfanthrene Brown G

Sulfanthrene Brown G Paste, a vat color of particular interest to the printing trade, has recently been announced by the Dyestuffs Department of E. I. du Pont de Nemours & Company. When printed, it is said to be bluer in shade than Leucosol Brown 3R Double Paste, and much redder than Ponsol Brown R Double Paste for Printing.

Besides its use for printing on cotton, rayon or silk, the product is claimed to be equally valuable for dyeing each of these fibers.

Fall Enrollment

Prospects for the fall enrollment of the Textile School of North Carolina State College are very encouraging, according to W. L. Mayer, director of registration, who stated that, judging from present indications, quite a number of States will be represented by students when college opens in September.

Dye Imports

Imports of synthetic dyes of coal-tar origin entered for consumption into the United States during the first six months of 1931 totaled 2,429,615 pounds valued at $2,113,623, increasing from 2,195,555 pounds invoiced at $1,839,417 in the corresponding half of 1930.

The rate for "Position Wanted" advertisements in this column is 2 cents a word—with a minimum of 50 cents per insertion. For all other types of advertisements—i.e., help wanted, machinery or supplies for sale—the rate is $5.00 per column inch or less per insertion.

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